ASSESSMENT OF SPONTANEOUS HEATING LIABILITY OF SOME INDIAN COALS USING EXPERIMENTAL TECHNIQUES

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF

BACHELOR OF TECHNOLOGY

IN

MINING ENGINEERING

BY

BIJAI SHANKER

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DEPARTMENT OF MINING ENGINEERING

NATIONAL INSTITUTE OF TECHNOLOGY

ROURKELA – 769008

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CERTIFICATE

This is to certify that the thesis entitled **"ASSESSMENT OF SPONTANEOUS HEATING LIABILITY OF SOME INDIAN COALS USING EXPERIMENTAL TECHNIQUES"** submitted by **Bijai Shanker (Roll no. 109MN0614)** in partial fulfillment of the requirements for the award of Bachelor of Technology degree in Mining Engineering at the National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

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Bijai Shanker

CONTENTS

ABSTRACT

Spontaneous combustion is the result of auto oxidation of coal. Majority of mine fire which occurs in the coalfields are mainly due to spontaneous heating of coal. Mine fire is a major problem for mining industry as well as the researcher in the field. It is important to determine the spontaneous heating susceptibility of coal for better production and minimization of the risk involved in the process.

The project deals with the various experimental investigations to find out the spontaneous heating susceptibility of some Indian coal. Eight coal samples for the project were collected from CCL and MCL mines, both from opencast as well as underground workings. The intrinsic properties as well as susceptibility indices of the coal samples were determined by following experimental techniques:

- Proximate analysis
- Calorific value
- Wet oxidation potential
- Crossing point temperature
- Olpinski index
- Flammability temperature
- Differential thermal analysis

From the experimental investigation, it is found that the coal samples collected from CCL coal fields as well as MCL coal fields are moderately susceptible to spontaneous heating of coal. Some of the coal samples are more susceptible than the other. But overall we can say that the coals are moderately susceptible to spontaneous heating.

LIST OF TABLES

LIST OF FIGURES

LIST OF PHOTOGRAPHS

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND

Coal mine fire is a major concern for the Coal mining industry. It has proved to be a major problem worldwide, especially in coal rich nations such as China. These fires have serious economic, social and ecological impacts. They are often started by lighting grass, or forest fires, and are particularly insidious because they continue to shoulder underground after surface fires have been extinguished. Coal fires are a serious health and safety hazard as well as affecting the environment by releasing toxic fumes, damaging machinery present inside the mine, reigniting grass, brush ,or afforest fires, and cause subsidence of surface infrastructure such as roads, pipelines, bridge supports ,electric lines, buildings and homes. Coal fires continue to burn for decades or even centuries in some cases, which are extremely difficult and costly to extinguish, as they burn underground [23].

All coals are not susceptible to spontaneous heating to the same extent and it is essential to assess the degree of proneness of coals in order to plan advance precautionary measures. Therefore, the determination of susceptibility of coals to spontaneous heating is essential to efficiently run the production and maintenance activities, so that required quantity of coal is available within incubation period. The spontaneous heating of coal is defined as the process by which coal starts burning by itself just by absorbing oxygen from atmosphere and not by any external fire. Some fraction of the exposed coal absorbs free oxygen at a faster rate than others which leads to oxidation with the formation of gases (CO,CO_2), water vapor and some heat during the reaction. The oxidation course of coal takes place even at ambient temperatures [24].

Now a day's various preventive technologies being adopted in order to determine the liability of coal to spontaneous heating different methods have been followed by various researchers of the world. A number of experiments have been performed and formulated for assessments of spontaneous heating susceptibility of coal viz., Differential thermal analysis, Crossing point temperature method, Olpinski index method , Wet oxidation potential technique, Flammability temperature. A number of approaches have been developed over the years to

assess the susceptibility of coal towards spontaneous heating. This liability to self-heating of coal also resolves the incubation period of coal seam, which decide the dimension of the panel to be created, which is a most vital safety should be taken in account while mine scheduling. It is therefore imperious that the persons making plan for a mine define in advance the spontaneous heating susceptibility of the seam to be mined so that either the entire coal has been taken out before the incubation period of the coal, or advance precautions are planned to tackle this problem and prevent spontaneous heating [22].

1.2 OBJECTIVE

The objectives of the project are as follows -

- **Literature review** – collection of all the past works done by various academicians/researchers/scientists both national and international.
- **Sample collection and preparation** – Five samples were collected from CCL and three from MCL, for the purpose of analysis and the samples were collected and prepared as per the Indian Standards.
- **Experimentation** – the experimentation part divided into two stages:
    1. Determination of intrinsic properties of coal – calorific value, proximate analysis.
    2. Determination of susceptibility indices of coal – Wet oxidation potential, Crossing point temperature, Olpinski index, Flammability temperature and Differential thermal analysis.
- **Analysis** - Correlation analysis was done using MS Excel package among the various parameters of proximate analysis and the susceptibility indices obtained to find out the best correlation among them. The effect of each parameter and indices are evaluated.

CHAPTER 2

LITERATURE REVIEW

2.1 NATIONAL AND INTERNATIONAL STATUS

The following is the brief review of the work carried out by different researchers to determine the spontaneous heating tendency of coal samples.

2.1.1 National status:

Banerjee et al. (1972) determined the Crossing Point Temperature (CPT) of a number of Indian coal samples adopting the Crossing Point Temperature method. He found that coals with crossing point temperatures above 160°C are badly susceptible. Between 120^0C & 140^0C could be considered to be extremely susceptible to spontaneous heating.

Nandy et al. (1972) illustrated the variation in Crossing Point Temperature values with the, oxygen percentage, moisture content and the volatile matter of coal. He detected that CPT normally minimizes with maximizes in each of these constituents of coals. But outside 35% V.M, 9% oxygen, or 4 to 6% moisture content there is not much change in CPT values. In fact, above 4 to 6% moisture content in coal, then CPT values shows a developing trend.

Banerjee (1985) derived the formula for the Olpinski index, in this method small pellet of coal is allowed to undergo aerial oxidation at temperature around 135^oC gives the measure of spontaneous heating susceptibility S_{Za} of the concerned coal. Olpinski method makes correction for ash content of the coal (A), and expressed spontaneous heating index as: $S_{Zb} = (S_{Za} / (100\text{-}A))*100)$

Tarafdar et. al (1987) reported results of wet oxidation of coal using alkaline permanganate solution involving measurements of differential temperature at different temperatures. This was done at a constant heating rate, and potential changes between a saturated calomel electrode and a carbon electrode immersed in the coal oxidant mixture within a definite reaction time at a constant temperature. Seven coal samples of known crossing point temperatures (CPT) were measured. Four samples, considered to be highly susceptible to spontaneous heating, had CPT in the range 132-137^0C, and three, considered poorly susceptible to spontaneous heating, had CPT values in the range 162-168^0C, which showed

two distinct zones of correlation between CPT values and the corresponding differential peak temperatures, and between CPT and the observed potential changes. They suggested that differential temperature and potential difference measurements during wet oxidation of coal may be used as alternative techniques for the assessment of tendency to spontaneous heating.

Panigrahi et. al (1996) carried out work on wet oxidation and CPT with respect to intrinsic properties of coal samples covering most of the coalfields of India and determine the susceptibility of coal to spontaneous heating.

Nimaje et. al (2010) made thermal studies on spontaneous heating of coal. Thermal studies play an important and dominant role among all the experimental techniques developed in assessing the spontaneous heating susceptibility of coal. They made an overview of thermal studies which was carried out by different researchers across the globe for determination of spontaneous heating of coal and showed that lot of emphasis on experimental techniques is necessary for evolving appropriate strategies and effective plans in advance to prevent occurrence and spread of mine fire.

2.1.2 International status:

Olpinski et. al (1953) observed the exothermicity of the coal pellet at 235^0C provides the spontaneous heating susceptibility of the coal and the electronic recorder recorded time-temperature curve of the coal bed crosses 235^0C. The rate of rise of temperature at 235^0C provides Olpinski index.

Feng et. al (1973) discovered a composite liability index using the results of Crossing Point Temperature experiment s, called FCC index. This is calculated using the following equation[19]

$$Liability\ Index = \frac{Average\ heating\ rate\ between\ 110°C\ and\ 220°C}{Relative\ investigation\ temperature} * 100$$

Gouws et. al (1988) gave three characteristics on a differential thermal analysis thermogram (i.e., the crossing-point temperature, stage II exothermicity gradient and the transition temperature to high-level exothermicity) are generally believed to be indicative of the self-heating susceptibility of coal. A new index was developed which was applied to 58 coals, which enabled known dangerous and safe coals to be identified [20].

Ren et. al (1998) used adiabatic calorimeter for the propensity of 18 pulverised coals (Australia, UK, US, Indonesia, South Africa, South America) to spontaneous combustion. All

the coal samples were tested at an initial temperature of 40°C and three samples at 60°C. Their propensities to spontaneous combustion were ranked according to their initial rate of heating (IRH) and total temperature rise (TTR) values. The results demonstrated that air humidity is an important factor is determined whether a heating will progress rapidly or not. The particle size distribution of the coal affects the IRH and TTR values, with relatively smaller particles tending to be more reactive. Aged and pre-oxidised coals have higher IRH and lower TTR values, and the coal becomes less reactive. The magnitude of the temperature raises (TTR) increases with increasing initial temperature.

2.2 MECHANISM OF SPONTANOEUS HEATING [1]

The auto oxidation of coal is a complex physio-chemical process which is consists of a series of event like oxygen absorption, coal-oxygen complexes formation and their decomposition leading to the discharge of heat. This complexity of the process is vast because of the great diversity in the coal substance with the associated mineral matter and the conditions of oxidation. During the oxidation of the heterogeneous mass, concurrent and overlapping reactions take place which are very difficult to separate out. The rate of oxidation at ambient temperature gives a measure of the proneness of coal to auto-oxidation. This low temperature aerial oxidation of coal is not a singular chemical process but a complex phenomenon generally comprising of several simultaneous and interesting chemical processes which result both in erosive material removal and structural alteration of the organic matter. A large number of stable chemical chain reactions take place due to several oxidation states of carbon and a variety of strong carbon oxygen complex is formed. The noticeable compositional, elemental and structural variations reveal that the reaction of oxygen with solid coal is a time dependent dynamic method. Noticeable variations in coal molecular structure and composition arise from prolonged sequence of events whose components exhibits complex inter-relationships. Thus the reaction environment is heterogeneous intrinsically because two bulk phases, solid and gas are present and extrinsically because various structural changes brought by the reaction, affect the overall coal reactivity. Porous solid absorbs the liquids or gases or the solution of gas/liquid, which is known as sorption. When accumulation restricts at the surface liberating heat and rate of penetration is negligible then it is considered as adsorption but if uniform penetration in the bulk of the solid occurs, then it is called absorption. The absorptions is always an endothermic phenomenon and starts from the surface of the solid and consumes heat of the solid for penetration. The energy on the surface is always low. The method where physical forces like inter-molecular attraction are blamable,

are known as physical adsorption or Van der Wall's adsorption, but when operative force needs to break the chemical nature of the compound is called chemisorption or activated adsorption. This physical adsorption is predominant at very low temp whereas chemical adsorption is very low at low temp whereas rate of reaction increases with increase in temperature. Coal gets heated up on absorbing oxygen, whose decomposition phenomenon can be expressed/explained in the following manner. Oxidation is very slow below 50^{o}C and accelerates above 50^{o}C, but above 80^{o}C, a period of steady state is maintained, probably due to the removal of moisture of coal. The removal of oxides of carbon occurs from 120^{o}C. The interaction of oxygen with coal accelerates rapidly up to 180^{o}C and thermal decomposition starts between 180^{o}C to 220^{o}C. Self-sustained process of combustion begins in between 220^{o}C to 275^{o}C with very rapid rise of temperature until the ignition point is attained.

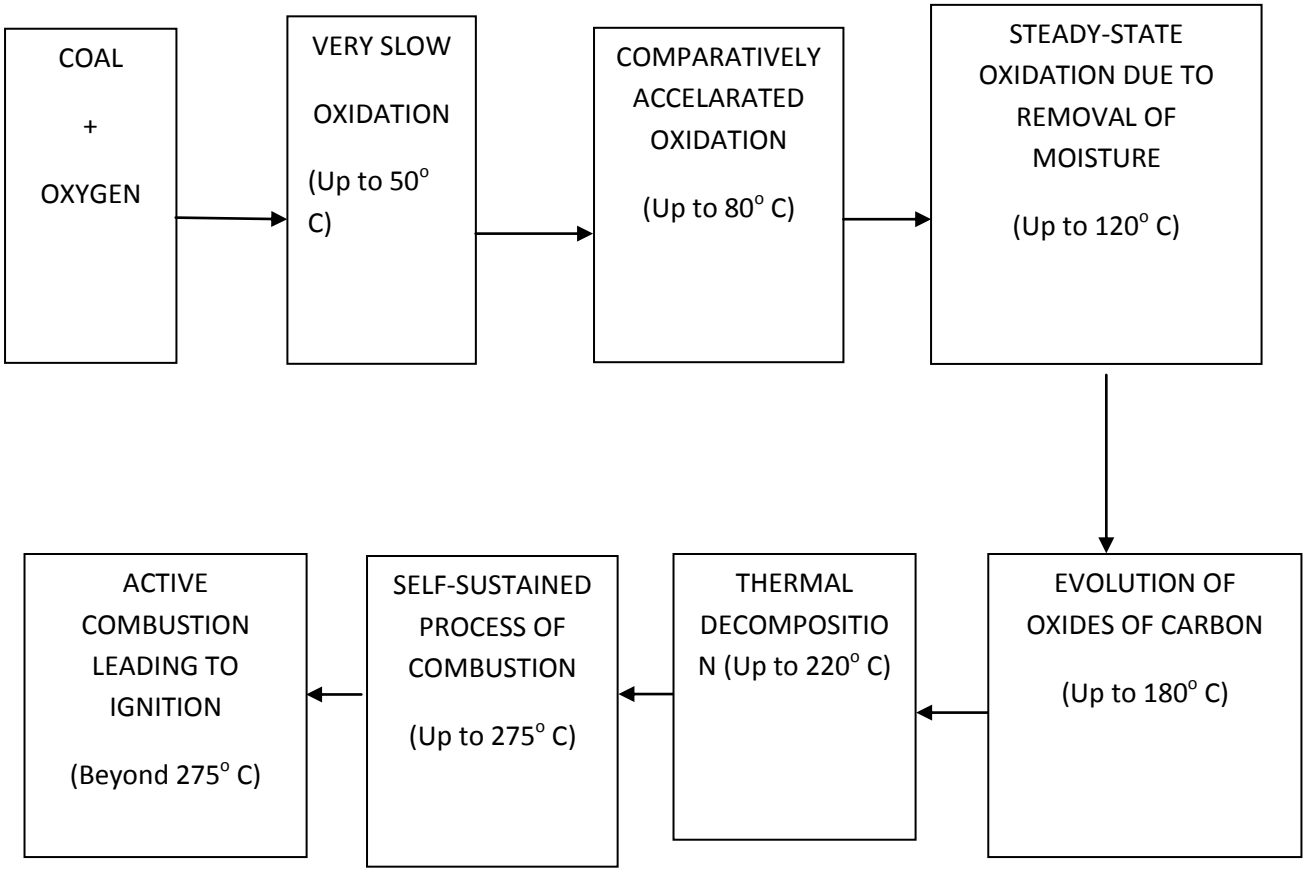

Fig. 2.1 Sequential stages in the spontaneous combustion of coal [1]

2.3 FACTORS AFFECTING SPONTANEOUS HEATING OF COAL[2]

The main reason for the difficulties in understanding the mechanism of spontaneous combustion is the presence of many internal and external factors which affects the initiation and development of the process. These factors are summarized below:

2.3.1 Intrinsic factors – These factors are mainly related to nature of coal

- Pyrites – As pyrite content increases the tendency of spontaneous heating increases.
- Inherent moisture – Changes in moisture content such as drying or wetting of coal have significant effects.
- Particle size and surface area – As particle size decreases the exposed surface area increases and the susceptibility increases.
- Rank and Petrographic constituents – Lower rank coals are more susceptible.
- Chemical constituents – Ash generally decreases liability for spontaneous heating but certain parts of ash such as lime and soda have accelerating effect whereas alumina and silica have retarding effects.
- Mineral matter – Some chemicals promote an others inhibit spontaneous heating.

2.3.2 Extrinsic factors – These are the factors which are mainly related to atmospheric, mining and geological conditions

- Temperature – Higher surrounding temperature leads to increase in oxidation process and ultimately in spontaneous heating of coal.
- Extraneous moisture – Evaporation of surface moisture leads to release of heat of wetting which adds to the temperature rise and increases susceptibility. The rate of oxidation is increased b the presence of atmospheric moisture
- Oxygen concentration – Higher the oxygen concentration in the atmosphere more rapid is the oxidation process as oxygen is readily available.
- Coal seam and surrounding strata – Presence of faults that lead to the passage of air and oxygen to the heating are generally increase the rate of heating.
- Method of working, ventilation and air flow rate – Mining methods such as bord and pillar mining that leave some pillars attracts more spontaneous heating than longwall methods. Air flow rate controls heating to a large extent. If the ideal flow

rate is maintained then it helps in dissipation of heat but if too much of air is flowing then it stagnates the heat and increases the heating.

- Timbering, roadways, bacteria and barometric pressure – Presence of timbers in the mines leads to the danger of catching of fire which gives the heat required for spontaneous heating of coal. Bacterial decomposition of coal and other wood products also releases some amount of heat which increases susceptibility.

Table 2.1 Factors affecting spontaneous combustion of coal [3]

Intrinsic Factors (Nature of Coal)	Extrinsic Factors (Atmospheric, Geological and Mining Conditions)
Pyrites	Temperature
Moisture	Moisture
Particle size and surface area	Barometric pressure
Rank and petrographic constituents	Oxygen concentration
Chemical constituents	Bacteria
Mineral matter	Coal seam and surrounding strata
Petrographic composition	Method of working
Volatile matter	Ventilation system and air flow rate
Rank of coal	Timbering
	Roadways

2.4 THEORIES OF SPONTANEOUS COMBUSTION OF COAL

2.4.1 Pyrite theory [1]

Combustion because of oxidation of pyrites has been recognized to be a common process in pyrite mines. Heating of coal can be initiated by iron pyrites (only when present in substantial proportion) and in finely powdered and spread state in the existence of moisture. The reaction between iron pyrites, oxygen and moisture is exothermic, hence yields materials of greater volume than the original pyrite thus availing more pore for oxygen to be absorbed. The reaction can be shown as

$$2Fe\,S_2 + 7O_2 + 16H_2O = 2\,H_2SO_4 + 2\,Fe\,SO_4.7H_2O + 316\ kcal$$

The oxidation reaction of pyrite during weathering of coal seam may be shown by

$$Fe\ S_2 + 15/4\ O_2 + 9/2\ H_2O = Fe\ O.OH + 2\ H_2SO_4$$

The equation shows that both oxygen and moisture, two main weathering causes, contribute to pyrite alteration and the sulphuric acid is made as a by-product of the change. In comparison to dry coals in the existence of moisture, the reactivity of coal is found to be double and if pyrite is in finely spread state, it becomes 10 fold. It was seen that pyrite below 5% showed worthless effect.

2.4.2 Bacterial Theory [1]

Bacteria were also assumed to encourage self-ignition of coal. Later examinations revealed that bacteria had little effect on the self-heating tendency of coal. The involvement of heating due to the action of bacteria cannot be totally ruled out. Spontaneous heating witnessed in haystacks and in wood are known to be mainly due to bacterial action. However, there is no vital proof to authenticate or cast out this theory.

2.4.3 Humidity theory [21]

It says the quantity of heat liberated by atmospheric oxidation of coal is much less than the amount of heat required removing water from the coal. If the evaporation of water can be induced at the seat of heating, then the temperature of heating would decline. When it is recalled that water is an oxidation product of low temperature oxidation of coal, the above system well explains other possible sources of CO and CO_2 in low temperature reaction between coal and oxygen. Nordon et. al (1979) measured the heat of wetting with water of an Australian low rank bituminous coal with different moisture content. Although dry coal showed a substantial rise of temperature, heat of wetting (6 KJ / Kg) values decreases rapidly with increasing moisture content and heat. For a normal moist coal (65% relative humidity) heat of wetting could cause a temperature rise of only 2°C, which would be unlikely to contribute importantly to self-heating in store coal.

2.4.4 Coal-oxygen Complex Theory [1]

Oxidation of coal is believed to be initiated at native radical sight. Formation of peroxy radical and hydro peroxides is commonly to be thought to be they mechanism by which oxygen and moisture are initially in corporate into organic matrix. These species may react, rearrange or decayed to form wide range of oxygen functionality in the matrix or gaseous product.

CHAPTER – 3

EXPERIMENTAL INVESTIGATION

To study the effects of various parameters of coal that affect the spontaneous heating tendency of coal, the following experiments were carried out:

- Proximate analysis
- Calorific value
- Wet oxidation potential
- Crossing point temperature
- Olpinski index
- Flammability temperature
- Differential thermal analysis

3.1 SAMPLE COLLECTION AND PREPARATION [4]

3.1.1 SAMPLE COLLECTION

It is the process by which the physical and chemical properties of the mineral or ore can be observed with the preferred accuracy. It is the method of collecting the small portions of a whole such that consistence of that portion presents that of entire mass .Various types of sampling are:

a. Channel sampling
b. Chip sampling
c. Grab sampling
d. Bulk sampling
e. Drill hole sampling

Out of the sampling methods mentioned above, channel sampling method is the common method generally accepted to collect the coal sample from the insitu coal seams following the Indian standard.

Channel sampling

The part of seam to be tested shall be exposed from the roof to the floor. The same sample shall be taken in a channel representing the entire cross-section of the seam having the

dimensions of 30 x 10 cm, that is, 30 cm in width and 10 cm in depth. For this purpose, two parallel lines, 30 cm apart at right angles to the bedding planes of the seam shall be marked by a chalked string on the smooth, newly exposed surface of the seam. Obvious dirt bands exceeding 10cm in thickness shall be excluded. The channel between the marked chalk lines in the seam shall be cut to a depth of 10 cm and the coal sample collected on a clean strong cloth or tarpaulin placed immediately at the bottom so that the chances of pieces flying off during excavation of coal are minimized.

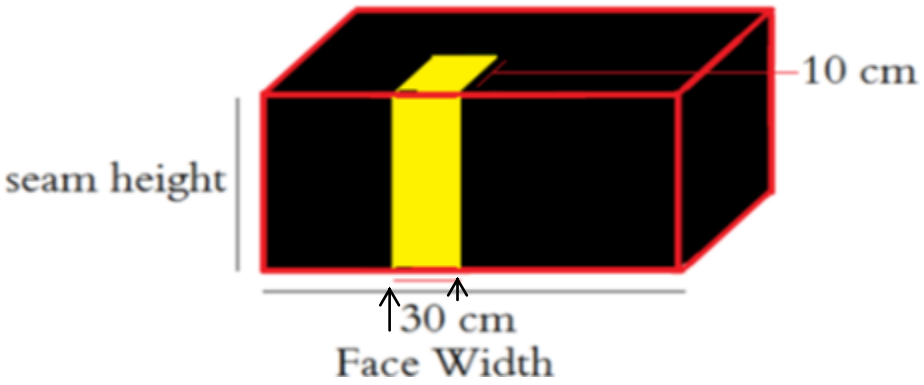

Fig. 3.1 Channel Sampling [5]

3.1.2 SAMPLE PREPARATION

The samples received from the field via channel sampling methods are crushed in the laboratory as per the experimental requirements. The crushed sample is then sieved to required sizes and stored in air tight polythene packets. The packets are stored in air tight containers for further use in experimentation. Samples were prepared according to IS 436 Part 1, Section 1-1964 and IS 436 Part II-1965.

3.2 PROXIMATE ANALYSIS [6]

Proximate analysis was developed as a simple mean of determining the distribution of products found of coal. When the coal sample is heated under specified conditions, then it classifies the products into four groups which are: i) moisture; ii) volatile matter iii) fixed carbon, iv) ash, the inorganic residue remaining after combustion. For proximate analysis of coal, the method determined by IS (Indian standard) 1350 (Part-I) – 1984 was followed.

3.2.1 Determination of moisture content (M)

Coal is always associated with some amount of moisture, which is both physically and chemically bound, due to its nature, origin and occurrence. It is customary to differentiate between extraneous and inherent moisture. When a wet coal is exhibited to atmosphere, the external moisture evaporates, but the obviously dry coal still contains some moisture.This can be removed by heating the coal above 100^0C. External moisture is also called accidental moisture and inherent moisture is called equilibrium or air-dried or hygroscopic moisture. The quantity of external moisture counts mainly on the handling of coal and the mode of occurrence whereas the air-dried moisture is associated to the inherent hygroscopic nature of the coal.

Procedure

1. About 1g of finely pulverized -212 micron air-dried coal sample is weighed in a silica crucible and then placed within an electric hot air oven and is maintained at 110^0C.
2. The crucible with the coal sample is allowed to put in the oven for 1.5 hours and it is taken out with the help of tongues. It is then then cooled in a desiccator for about 15 minutes.
3. Then weighed and loss in weight is reported as moisture (on percentage basis).
4. The calculation is done as per the following.

$$\textbf{Moisture } \% = \frac{\mathbf{Y - Z}}{\mathbf{Y - X}} * \mathbf{100}$$

Where,

X - Weight of empty crucible, in grams (g)

Y - Weight of crucible and coal sample before heating, in grams (g)

Z - Weight of crucible and coal sample after heating, in grams (g)

3.2.2 Determination of volatile Matter (VM)

When coal is heated in defined equipment under appointed conditions, is concerned to as volatile matter, the loss of mass and corrected for moisture. The matter lost is composed of materials that form upon the thermal decomposition of the various constituents of coal. Some of the element of coal volatile matter is hydrogen, carbon monoxide, methane and other hydrocarbons, tar vapors, ammonia, some organic sulphur and oxygen containing deepens and some incombustible gases, such as carbon dioxide and water vapour.These are formed as a result of the decomposition of organic materials present in the coal in coal. Inorganic

materials in coal contribute the water of hydration of mineral matter, carbon dioxide from carbonates and hydrogen chloride from inorganic chlorides to the volatile matter.

Procedure

Determation of the volatile matter was done using a volatile matter silica crucible (38 mm height, 25 mm external diameter and 22 mm internal diameter).

1. First the empty silica crucible along with the lid uncovered was heated at 800^0C for an hour in the muffle furnace and then cooled to room temperature.
2. The empty volatile matter crucible was then weighed again.
3. Approximately 1g of coal sample was weighed in the volatile matter crucible and it was placed inside the muffle furnace maintained at 925^0C with the lid covering the crucible.
4. The heating was carried out for 7 minutes, and then the crucible was removed.It was then cooled in air and a desiccator and weighed again.

$$\textbf{Volatile matter}\ \% = \frac{Y - Z}{Y - X} * 100 - M\%$$

Where X = weight of empty crucible, in grams (g)

Y = weight of crucible and coal sample before heating, in grams (g)

Z = weight of crucible and coal sample after heating, in grams (g)

M%=Moisture percentage

3.2.3 Determination of Ash (A)

During the ashing process, the coal ash is the residue left after the combustion of coal under defined conditions. Ash is formed as the result of chemical changes that take place in the mineral matter.The extraneous and inherent mineral matters are the two types of ash forming materials in coal. The extraneous mineral matter consists of materials like calcium, magnesium and ferrous carbonates, pyrite, marcasite, clays, shales, sand and gypsum.

Procedure

1. Weight of the empty crucible is taken.

2. 1g of desired coal sample is weighed in the crucible and is taken in a muffle furnace at 450^0C 3. For 30 minutes and the temperature of the furnace is raised to 850^0C for 60 minutes.

4. After that time interval, the crucible is taken out and placed in a desiccator and weighed.

$$\textbf{Ash}\% = \frac{Z - X}{Y - X} * 100$$

Where,

X= weight of empty crucible in grams (g)

Y= weight of coal sample and crucible in grams (g) before heating

Z= weight of coal sample and crucible in grams (g) after heating

3.2.4 Determination of Fixed Carbon (FC)

Fixed carbon is the materials remaining after the determination of moisture, volatile matter and ash. Fixed carbon plus ash present the approximate yield of coke from coal. The value of fixed carbon is determined by subtracting from 100 the resultant summation of moisture (M), volatile matter (VM) and ash (A).With all portion on the same moisture reference basis.

FC=100-(M+VM+A)

3.3 Calorific value [15]

The calorific value or heat of combustion is defined as the amount of heat evolved when a unit weight (or volume in the case of a sample of gaseous fuels) of the fuel is completely burnt and the products of combustion cooled to a standard temperature of 298^{o}K.

Procedure:

- About 1 g of coal sample is taken in a pellet press and a pellet is formed of nearly 1 g weight.
- The pellet is put in the lid provided and the nickel wire is put on.
- A thread is suspended from the nickel wire that is in direct contact with the coal pellet.
- The bomb is then closed by putting on the lid firmly from the top.
- Oxygen is supplied into it till the pressure inside the bomb is 30 bar.
- 1 kg of water is put into the vessel provided and the bomb is put in the stand so that the top of the bomb is in layer of water level.
- The overall lid is closed and the stirrer is switched on along with the digital thermometer.
- The stirrer is allowed to run till the temperature attains a near constant value.
- The cord is put into the furnace and the bomb is fired after the attainment of constant value.
- The temperature rises initially at a higher rate and later settles down at a constant value.
- The initial and final temperature is noted.

- The water equivalent of the instrument is noted.
- The calorific value of coal is calculated by using the relation below :

Calorific value = $((T_f - T_i) * \text{Water equivalent}) / \text{Weight of the pellet}$

Where,

T_f – Final temperature

T_i – Initial temperature

Useful heat value = 8900 - 138(A+M)

Plate 3.1 Bomb calorimeter

3.4 Wet oxidation potential [11, 12, 13, 14]

The coal molecule may consist of two parts: one is condensed aromatic structure, which are resistant to oxidation and other one is the aliphatic or hydro aromatic structure that are more prone to oxidation .Coal structure becomes sensitive to oxidation due to the presence of hydroxyl group in the aromatic structure and this property becomes the cause for low rank coals because low rank coals carry more branch aliphatic hydro-carbons. Since the high rank coals possess structure very similar to that of graphite, it is less prone to oxidation. Lower rank coals on being oxidized produces large amounts of aliphatic acids compared to higher rank coals. When coal is made to react with alkaline permanganate, oxidation takes place and the concentration of magnetite ion in solution tends to increase comparative to permanganate and there will be outcome change in the potential till all the oxidation possible in coal molecule is finished. Thus it is observed that, addition of coal to alkaline permanganate solution results in a change of potential of carbon electrode sank in the solution. It is based on the chemical reaction of coal sample with solution of $KMnO_4$ and KOH.

The solution of alkaline potassium permanganate and potassium hydroxide with coal sample forms an electrochemical cell which on stirring produces EMF against a standard potential of 0.56V. The plot of the EMF's versus time gives an idea of the propensity of the coal sample towards spontaneous heating.

Plate.3.2 Wet oxidation potential apparatus

Principle of Wet oxidation

In wet oxidation process strongly alkaline solution of potassium permanganate ($KMnO_4$) is taken. The permanganate ion undergoes one electron reduction to magnate ion by the following reaction:

$$MnO_4^- + e \rightarrow MnO_4^{2-}$$

The standard electrode potential of this redox couple (E^0) is 0.56V. The electrode potential (E) is related to the concentrations of magnate and permanganate ions present in the solution and is given by the following equation:

$$E = E^0 - \frac{RT}{F} \ln \frac{[MnO_4^{2-}]}{[MnO_4^-]}$$

Where,

R = Universal gas constant

T = Temperature

F = Faraday's constant

When coal is added to alkaline permanganate, oxidation takes place and the concentration of magnate ion in solution increases relative to permanganate and there will be resultant change in the potential till all the oxidation possible in coal molecule is complete. Therefore, addition of coal to alkaline permanganate solution results in a change of potential of carbon electrode dipped in the solution. The electrode can be represented as carbon/Mno_2^-

Procedure:

The beaker along with the electrodes was placed over a magnetic stirrer such that homogeneity of the mixture of coal and alkali solution is maintained. The Teflon coated fish of the magnetic stirrer was placed inside the beaker. 0.5 g of coal sample of -212 (μ) sizes was mixed with 100 ml of deci-normal solution of potassium permanganate ($KMnO_4$) in 1N potassium hydroxide (KOH) solution in a beaker and the coal sample was subjected to wet oxidation process. The coal-oxidant suspension was continuously stirred using the magnetic stirrer. The potential difference (EMF) was recorded between the calomel and carbon electrodes over a period of time by using a millivolt meter till the potential difference attained a nearly constant value.

The graphs between Times vs. EMF (millivolt) for all the samples are plotted in appendix-1. Different samples require different time duration for attaining a nearly constant potential difference (ΔE). It was observed that almost all the coal samples attained a constant value after 30 minutes. Therefore this time period was chosen to find out the wet oxidation of the collected coal sample carried out at low, medium and high speed of the magnetic stirrer and the result are illustrated in Table 4.4.

3.5 CROSSING POINT TEMPERATURE METHOD

This is one of the oldest approaches for determining susceptibility towards spontaneous heating of coal. It is the temperature at which the coal temperature coincides with that of the furnace temperature or bath temperature in °C. In this method, the coal sample is heated in a furnace within a reaction tube at constant rising temperature with oxygen passing through it at a predetermined rate till the coal temperature crosses the furnace temperature [7].

Procedure [1]

The setup for the determination of crossing point temperature (CPT) of coal consists of following: Vertical tubular furnace which has an internal diameter of 50 mm and a heating capacity of 3KW. The furnace is provided with a temperature controller. Glass reaction tube is of 26 mm internal diameter and 150 mm in length. The reaction tube has spiraling glass tube of 6 mm internal diameter around it which is connected to the bottom (inside) of the reaction tube for air inlet and a small out-let tube at the top acts as air/gas outlet.

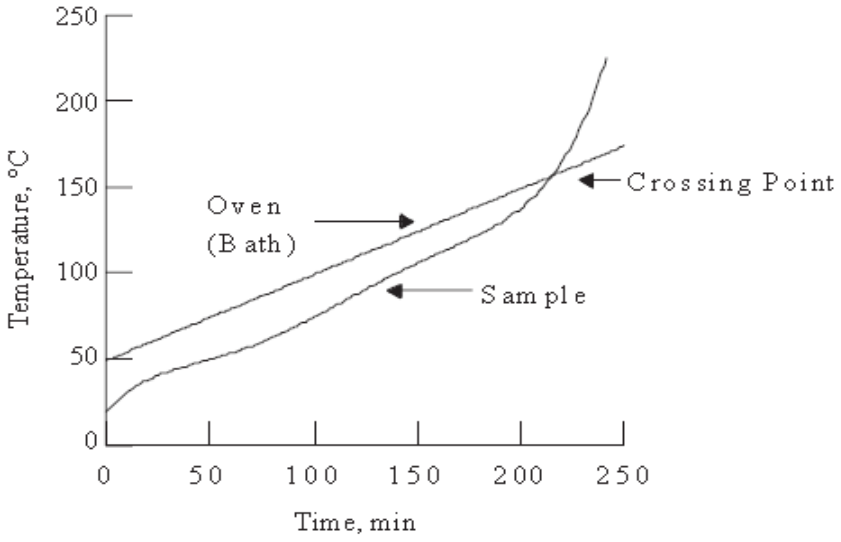

Fig. 3.2 Time Vs. Temperature curve of CPT [3]

A sulphuric acid bubbler is used to remove moisture in air. A drying tower containing granular calcium chlorides is used to remove moisture from air. 4g sample of size -100+200 mesh (Dp = 112 micron) was placed in the reaction tube followed by glass wool at the bottom most position and a small sieve of 200 mesh (Dp = 72 micron) on that. The tube is then lightly tapped a fixed number of times to achieve uniform packing density of the samples. The reaction tube is then placed in the tubular furnace and a chromel-alumel thermocouple is inserted at the center of the sample. The entrapped air and occulted gases are removed from the coal samples by passing a mild current of nitrogen through the sample for about three minutes, without disturbing the packing. The furnace is switched on and oxygen is allowed to pass through the sample simultaneously, with an average heating rate of 1^0C per minute and at 80 ml/ min. the Temperature of the furnace (bath) and the coal sample are recorded at every five minute interval till the temperature of coal crossed over and gone beyond the furnace temperature.

Table 3.1 Classification of CPT [8]

CPT(^{0}C)	Risk Rating
120-140	Highly susceptible
140-160	Moderately susceptible
>160	Poorly susceptible

Plate. 3.3 Crossing point temperature set-up

3.6 **OLPINSKI INDEX METHOD [9]**

In this method, liquid quinoline is heated in an electric oven to boil gently at a temperature of 230^{0}C producing quinoline vapour. The coal samples are powdered and small pellets of 1g of -72 mesh are prepared out of these powdered samples. These pellets are heated indirectly by quinoline vapour in an atmosphere of oxygen which is made to flow over the coal pellet at predetermined rate. While heating the coal pellets, a thermocouple is inserted into it and output of the thermocouple is given to a temperature recording system. The temperature versus time plot of the coal samples is directly obtained by this instrument. The rate of rise of temperature at the moment of equalization of supplied oxygen gas and coal pellet temperature is either graphically determined by drawing tangent to the curve at the point corresponding to the quinoline vapour temperature at 230^{0}C or the first derivative of the curve at 230^{0}C represents the slope of the curve at the same temperature. The rate of rise of temperature determined in this way is expressed in ^{0}C/min is an indication of spontaneous heating susceptibility of the coal .This index is known as Olpinski index and is denoted by Sz_a. In this

method Sz_a index is corrected for ash content of the coal and is expressed as Sz_b which is as given below:

$$Sz_b = (Sz_a\ (100\text{-}A))*100$$

Where, Sz_b – Spontaneous heating index free of ash

A – Ash content of coal expressed in %.

The increase of Sz_b index indicates that the sample is more susceptible to spontaneous combustion.

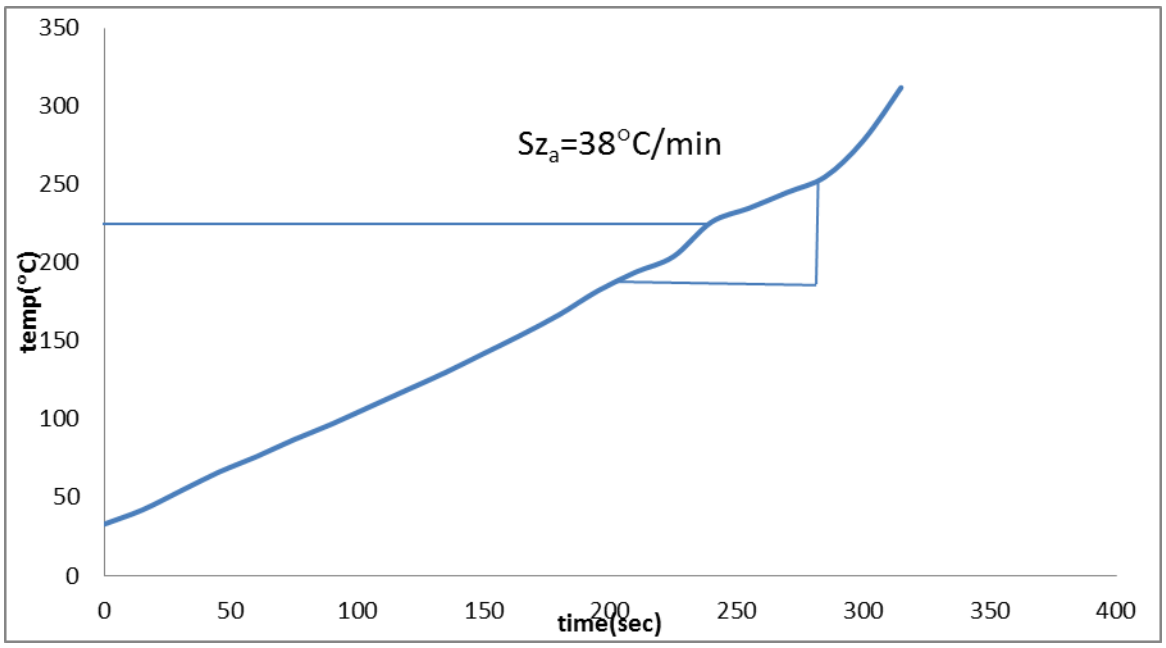

Fig 3.3 Olpinski curve

Plate. 3.4 Olpinski index apparatus

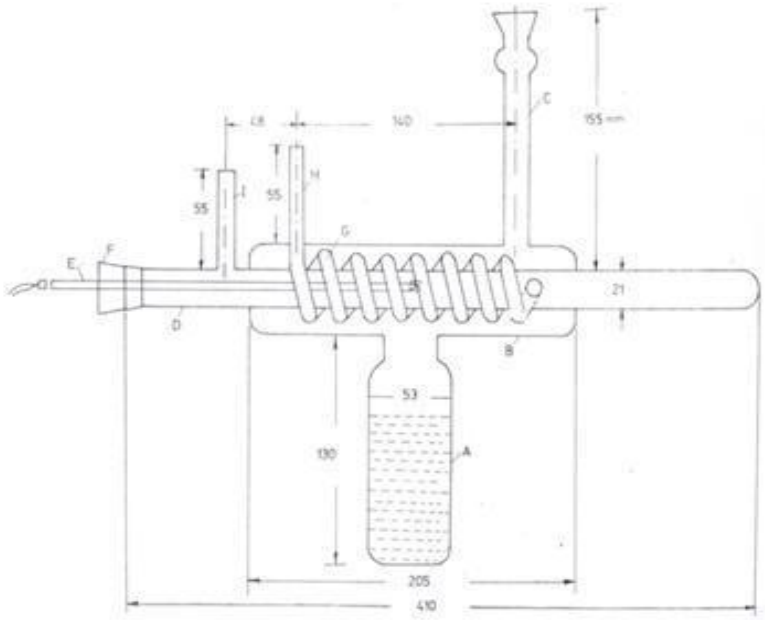

Fig. 3.4 Schematic diagram of Olpinski index apparatus [10]

The apparatus consists of a cylindrical glass vessel (A) of volume about 280 cc which is connected to another cylinder (B) perpendicular to it. One funnel (C) is attached to B which also acts as a condenser. A reaction tube (D) passes axially through B which can be heated by quinoline vapour. One end of a thermocouple (E) can be introduced to reach up to the central line of quinoline vessel. A glass tube (G) goes spirally over the reaction tube to release at its closed end. The combustion product goes out through the outlet (I). The apparatus is mounted over the electric oven to the heat quinoline vessel. The whole set-up placed in a fume chamber with an exhaust fan to clear the quinoline vapour out of the laboratory [9.]

Table 3.2 Classifications of Olpinski index [9]

Sz_a ($_0$C/min)	**Risk Rating**
>180	Very Highly Susceptible
80-180	Highly susceptible
40-80	Moderately susceptible
0-40	Poorly susceptible
<80	Poor liability to S.H. risk
>120	High liability to S.H. risk
80-120	Moderate liability to S.H. risk
39-193 °C/min For Indian coals	

3.7 Flammability temperature method [11]

The set up for the determination of the flammability temperature of coal consists of vertical tubular furnace having internal diameter 50mm and length 300mm.It is open at both ends and consists of a dust dispersing unit, a solenoid valve, a reservoir for passing of air, a mercury manometer, a drying tower and an aspirator bulb. In the helical dust disperser coal dust sample is kept. Air from the reservoir is allowed to pass through the disperser and on emergency from the divergent mount, forms a uniform dust-air mixture inside the furnace. The minimum temperature at which this mixture catches fire, is the flammability temperature of the coal dust. This is indicated by the appearance of flame coming out of the bottom of the tubular furnace

Procedure:

- Place 200 mg sample of coal having mesh size -200 mesh (BSS) in a helical tube**.**
- Maintain mercury column difference of 8 cm by aspirator bulb and turn off the tap.
- Switch on the solenoid valve at desired temperature of furnace, which permits the air to pass through it very fast and find out the status of coal sample (spark, smoke or flame).
- If flame appears then find out the exact temperature in lower temperature range. This is done by trial and error method. If the flame do not appear go for higher temperature range.

Plate 3.5 Flammability temperature apparatus

3.8 Differential thermal analysis [11]

It is often used to determine the physical property of a substance as a function of temperature. This method analyses the effect of temperature on the properties of the sample and compares it with an inert reference material which is usually alumina. Both are subjected to identical heat treatments and the temperature difference between sample and inert reference is measured and time vs temperature is plotted. The DTA apparatus consists of a sample and reference holder, a furnace, a temperature programmer to which maintains a constant temperature rate and an output which monitors the test performed. The sample holder is provided with two thermocouples each for reference and sample. The sample is contained in a small crucible. The direct contact of the thermocouple with the sample should be avoided. By the temperature programmer the temperature is made to rise at a constant rate and the temperature difference of the sample and reference is plotted against time. The plot consists of three parts – stage I, II, III.

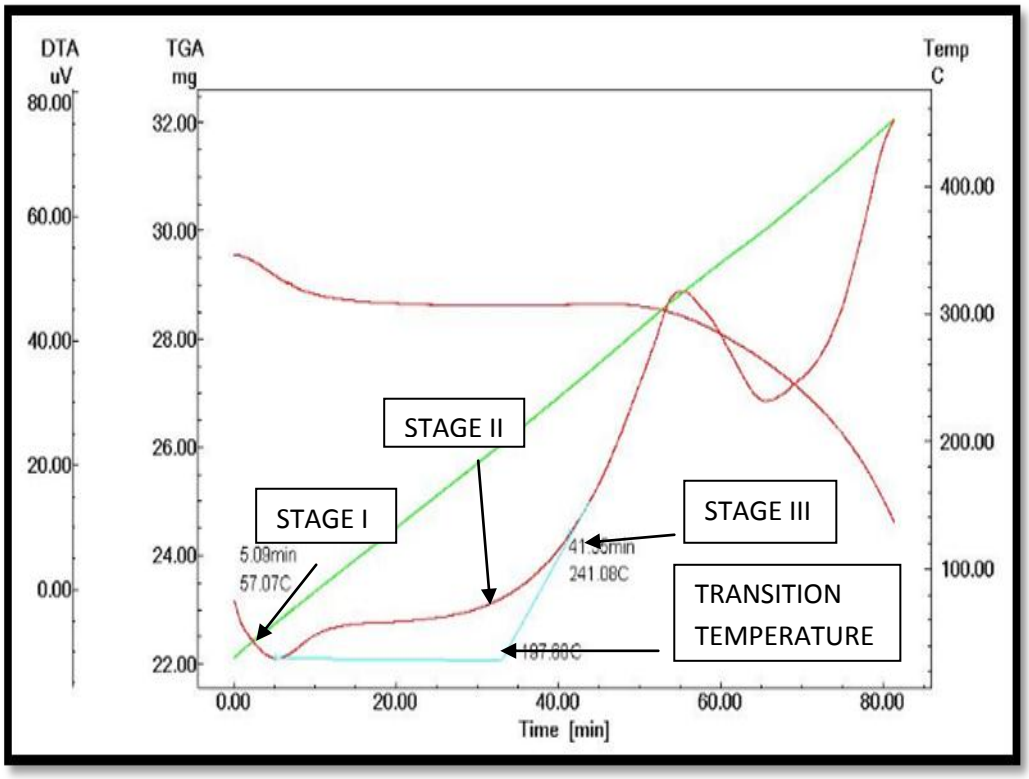

Fig 3.5 Different stages of DTA

Stage I mostly endothermic reaction takes place and the temperature falls due to the release of moisture.

Stage II consists of two parts – II A and II B. The heating tendency starts from the beginning of II A which accompanied by a small amount of endothermic reactions. The point where II

A begins is called as the inflexion point as the thermo gram suddenly rises. In II B exothermic reactions start to dominate.

The beginning of stage III is called as Transition temperature. This temperature is very important as it is directly related to spontaneous heating tendency of coal. The lower the transition temperature the higher is the susceptibility of coal and vice versa. From stage III onwards complete exothermic reactions occur and the temperature continues to rise steeply.

From the thermo gram plot four important points are required – slope of II A, II B, overall slope of II and the transition temperature. Lower the slope values lower is the susceptibility. The transition temperature is found out by drawing tangents at the inflexion point and any point on stage III. Their intersection gives the transition temperature.

Procedure:

- About 10 mg of -212 micron (BSS) coal sample is taken in the crucible.
- The reference material taken is alpha alumina.
- The DTA machine is switched on and the software is set according to required conditions viz., heating rate is maintained at 50 C/minute and the final temperature is taken as 4500C.
- The plots obtained are then analyzed for the slopes of various stages and transition temperature.

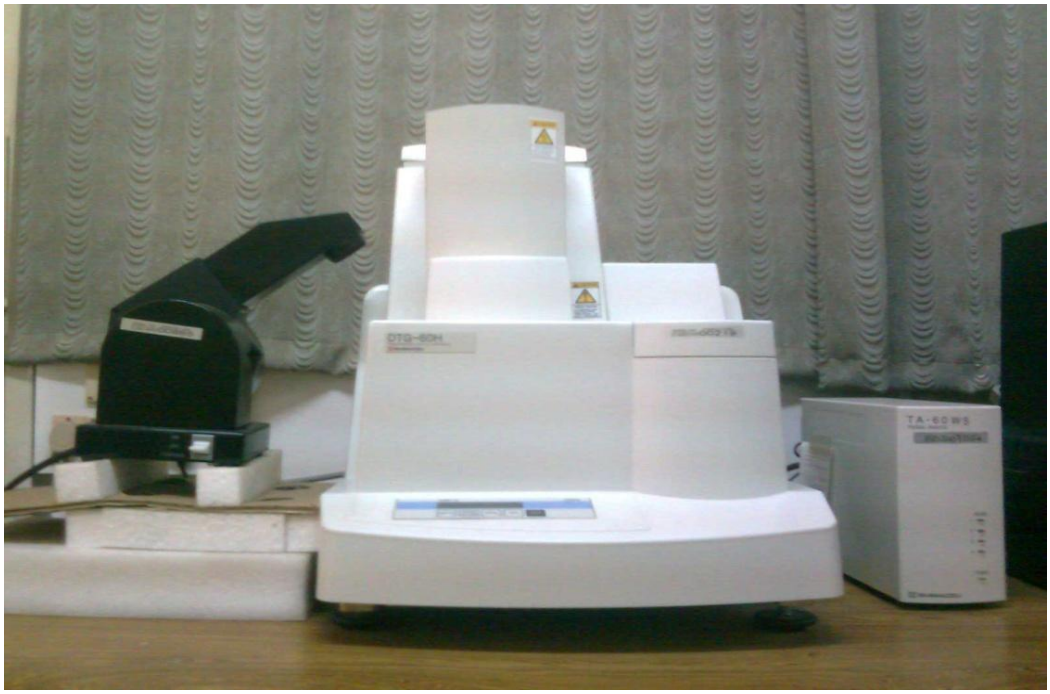

Plate 3.6 DTA-TG apparatus

CHAPTER- 4

RESULTS AND ANALYSIS

4.1 RESULTS

The eight coal samples were collected from different coalfields of India, five from CCL and three from MCL and are listed in the following table.

Table 4.1 List of coal samples

Sr. No.	Name of the Coal Sample	Name of the Coalfield
1	CCL-1	CENTRAL COALFIELD LIMITED
2	CCL-2	
3	CCL-3	
4	CCL-4	
5	CCL-5	
6	MCL-1	MAHANADI COALFIELD LIMITED
7	MCL-2	
8	MCL-3	

The results of the parameters of proximate analysis (Moisture, Volatile matter, Ash and Fixed carbon) after removing the extraneous moisture are calculated using the standard procedure of all the collected coal samples covering some coalfields of India and are listed in Table 4.2

Table 4.2 Results of the parameters of proximate analysis

Sr. No.	SAMPLE	M (%)	VM (%)	A (%)	FC (%)
1.	CCL-1	6.20	28.90	34.45	30.45
2.	CCL-2	6.20	27.35	37.00	29.45
3.	CCL-3	4.85	26.55	27.40	41.20
4.	CCL-4	5.60	25.70	30.20	38.50
5.	CCL-5	5.85	30.10	32.75	31.30
6.	MCL-1	7.20	41.75	12.45	38.60
7.	MCL-2	6.30	39.05	16.65	38.00
8	MCL 3	8.25	37.95	18.15	35.65

Results of the gross calorific values of all the collected coal samples using bomb calorimeter are listed in the following table 4.3

Table 4.3 Results of Calorific value

Sr. No.	Sample	Gross Calorific Value(Kcal/Kg)
1	CCL-1	4656.69
2	CCL-2	5111.33
3	CCL-3	4848.65
4	CCL-4	5538.50
5	CCL-5	4719.28
6	MCL-1	4715.82
7	MCL-2	5793.14
8	MCL-3	6096.49

Results of the wet oxidation potential difference of all the collected coal samples using wet oxidation apparatus are listed in the following table 4.4

Table 4.4 Results of the wet oxidation potential difference

Sr. No.	Sample	Wet Oxidation Potential Difference (mV)
1	CCL- 1	83.95
2	CCL- 2	79.60
3	CCL- 3	76.60
4	CCL- 4	84.35
5	CCL- 5	77.25
6	MCL- 1	98.10
7	MCL -2	108.70
8	MCL-3	118.10

The crossing point temperatures of all the coal samples are determined using the CPT apparatus designed by Prof Ramlu in an air medium bath and the results are listed in the following table 4.5

Table 4.5 Results of CPT

Sr. No.	Sample	CPT(°C)
1	CCL-1	158
2	CCL-2	161
3	CCL-3	164
4	CCL-4	159
5	CCL-5	167
6	MCL-1	166
7	MCL-2	176
8	MCL-3	162

The olpinski index of all the coal samples are determined by using the standard Olpinski index apparatus and the results are listed in the following table 4.6

Table 4.6 Results of Olpinski index

Sr. No.	Sample	Sz_a (°C/min)
1	CCL-1	52
2	CCL-2	40
3	CCL-3	36
4	CCL-4	42
5	CCL-5	34
6	MCL-1	46
7	MCL-2	32
8	MCL-3	38

The flammability temperature of all the coal samples are determined by using flammability temperature apparatus and the results are listed in the following table 4.7

Table 4.7 Results of flammability temperature

Sr. No.	Sample	Flammability temperature(ºC)
1	CCL-1	530
2	CCL-2	515
3	CCL-3	535
4	CCL-4	520
5	CCL-5	510
6	MCL-1	540
7	MCL-2	560
08	MCL-3	580

The transit temperature and the slope of all the coal samples are determined by using DTA apparatus and the results are listed in the following table 4.8

Table 4.8 Results of DTA

Sr. No.	Sample	Transition temperature (°C)	Slope II	Slope IIA	Slope IIB
1	CCL-1	184.69	0.0834	0.0631	0.1286
2	CCL-2	209.71	0.0791	0.0603	0.1247
3	CCL-3	203.88	0.0821	0.0674	0.1257
4	CCL-4	196.21	0.0744	0.0569	0.1225
5	CCL-5	197.60	0.0941	0.0832	0.1393
6	MCL-1	176.65	0.0851	0.0643	0.1304
7	MCL-2	181.27	0.0743	0.0537	0.1231
8	MCL-3	177.98	0.0962	0.0843	0.1403

4.2 CORRELATION ANALYSIS

Correlation analysis was done using the MS Excel 2007 package. Microsoft Office Excel 2007 provides powerful tools and features that one can use to analyze, share, and manage data with ease. Commands and features that were often buried in complex menus and toolbars are now easier to find out task-oriented tabs that contain logical groups of commands and features. The following table shows the correlation between the parameters of proximate analysis and WOPD.

Table 4.9 Results of correlation analysis of coal samples

SI → / IP ↓	WOPD	CPT	Sz_a	FT	II	IIA	IIB	Tr
M	0.82	0.02	0.16	0.44	-0.21	-0.24	-0.10	-0.72
VM	0.83	0.59	-0.09	0.57	0.32	0.26	0.38	-0.84
A	-0.79	-0.56	0.17	-0.38	-0.16	-0.14	-0.20	0.78
FC	0.27	0.32	-0.26	-0.13	-0.07	-2.13	-0.29	-0.23
CV	0.75	0.21	-0.43	0.70	-0.28	-0.20	-0.17	-0.30

Where,

M – Moisture

A – Ash

VM – Volatile matter

FC – Fixed carbon

WOPD-Wet oxidation potential difference

CPT-Crossing point temperature

Sz_a-Olpinski index

FT-Flammabiity temperature

CV-Calorific value

Tr-Transition temperature

SI-Susceptibility indices

IP-Intrinsic properties

FINDINGS

1. Moisture shows good positive correlation with wet oxidation potential and poor correlation with Sz_a and FT.
2. Moisture and CPT have very poor positive correlation.
3. Volatile matter shows good positive correlation with wet oxidation potential, CPT and FT, but shows poor negative correlation with Sz_a.
4. Ash content shows good negative correlation with wet oxidation potential, CPT and poor negative correlation with FT, but Ash shows poor positive correlation with Sz_a.
5. Fixed carbon shows poor positive correlation with wet oxidation potential and CPT, but shows poor negative correlation with Sz_a and FT.
6. Volatile matter shows positive correlation with slope II,IIA and IIB obtained from the DTA thermo gram
7. Ash content shows good positive correlation with transition temperature

CHAPTER- 5

CONCLUSIONS

Samples of eight different mines from two coal fields were analyzed. The results obtained from the correlation of proximate analysis with wet oxidation potential, CPT, Olpinski index and Flammability temperature concludes the following:

- Highest moisture content was found in MCL-1 and MCL-3, it was also found during field observation that the seam was watery.
- Results of CCL-1 and CCL-4 shows CPT below 160°C and Sz_a nearby to 40°C/min, so these are moderately susceptible to spontaneous heating.
- Samples CCL-2, CCL-3, CCL-5, MCL-1, MCL-2 and MCL-3 have CPT above 160°C and Sz_a over 40°C/min, which implies that these are poorly susceptible to spontaneous heating. CCL-1 has the highest Oplinski index and lowest CPT, therefore it is more susceptible to spontaneous heating than other samples.
- CCL-3 has lowest wet oxidation potential, thus indicating low susceptibility towards spontaneous heating.
- DTA results show that MCL-3 has highest slope and lowest transition temperature, thus it has high susceptibility towards spontaneous heating.

Based on the above finding and the correlation analysis, the fire risk of the corresponding coal samples are listed in the following table:

Table 5.1 fire risk of the collected coal samples

Sr. No.	Name of the Coal Sample	Fire risk
1	CCL-1	Medium
2	CCL-2	Low
3	CCL-3	Low
4	CCL-4	Medium
5	CCL-5	Low
6	MCL-1	Medium
7	MCL-2	Low
8	MCL-3	Medium

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APPENDIX-1

WET OXIDATION POTENTIAL CURVES

The graphical presentations of all the wet oxidation curves are shown below:

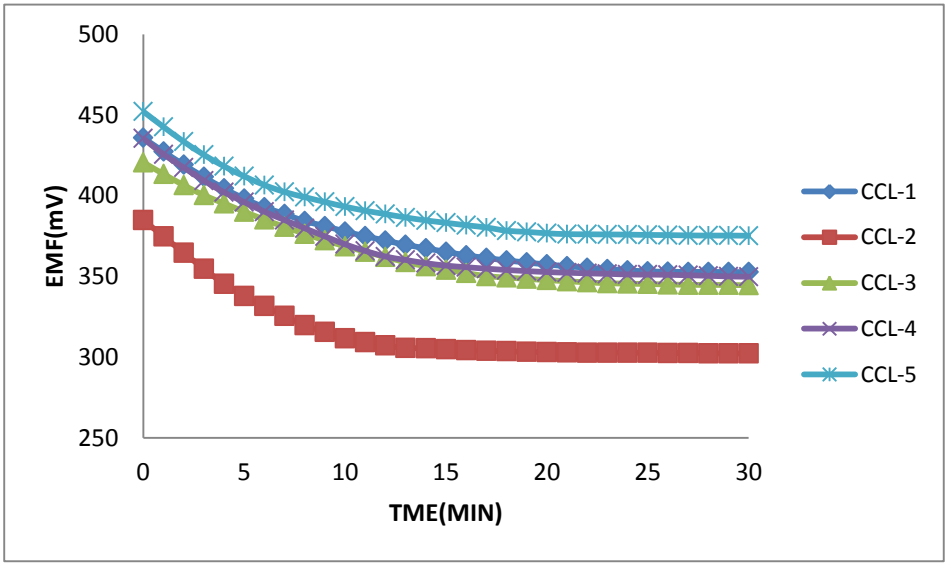

Fig. A-1 Wet oxidation potential curve of CCL coals.

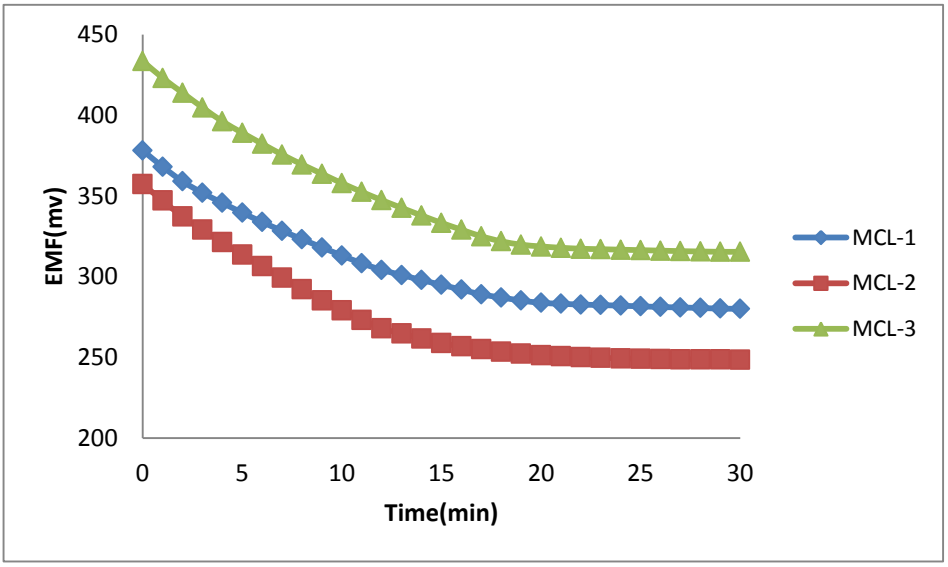

Fig. A-2 Wet oxidation potential curve of MCL coals.

APPENDIX-2

CPT CURVES

The graphical presentation of crossing point temperature of all the coal samples is shown in the following figures:

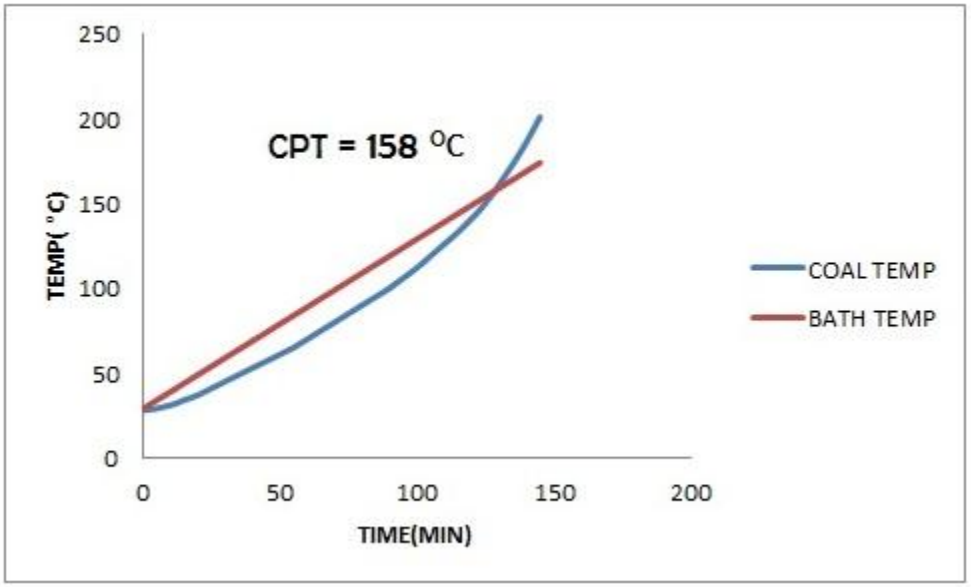

Fig. B-1 CPT curve of CCL-1

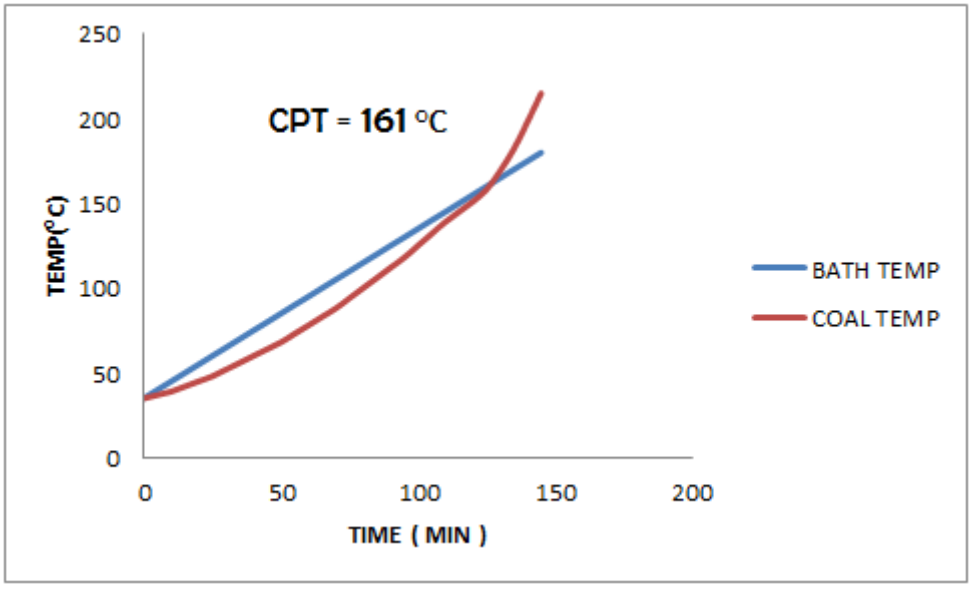

Fig. B-2 CPT curve of CCL-2

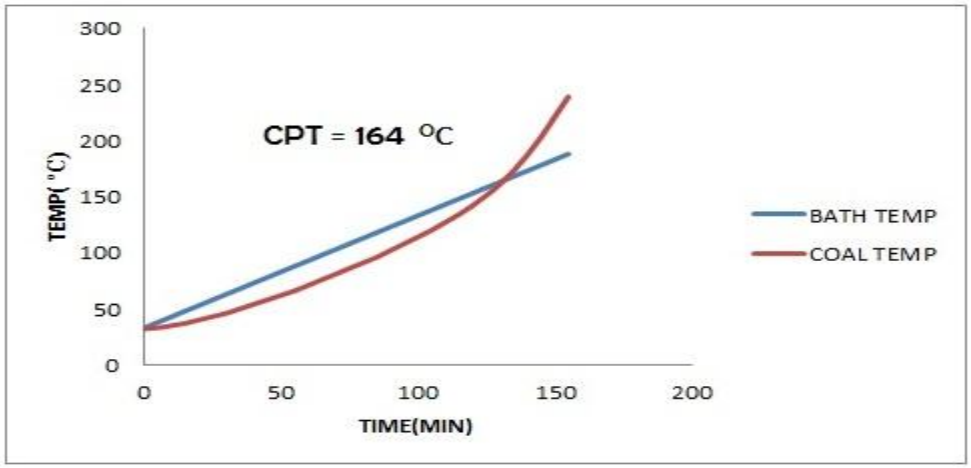

Fig. B-3 CPT curve of CCL-3

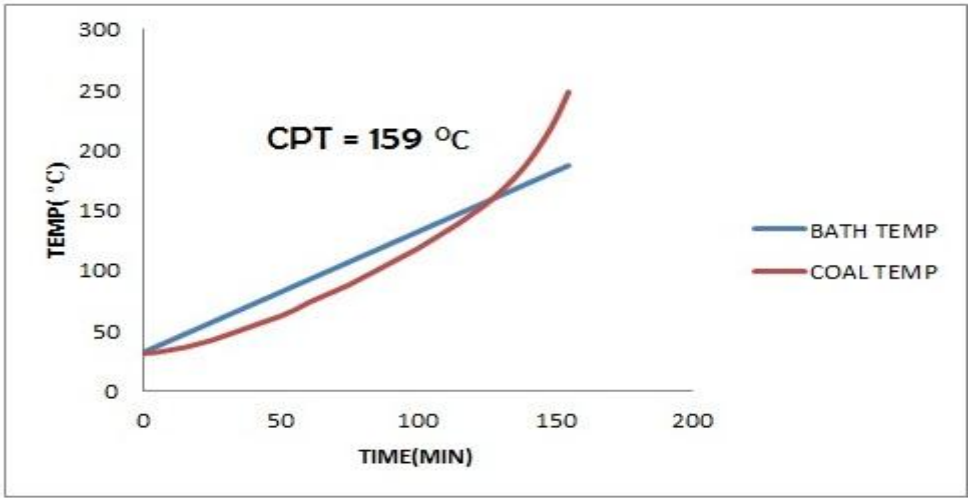

Fig. B-4 CPT curve of CCL-4

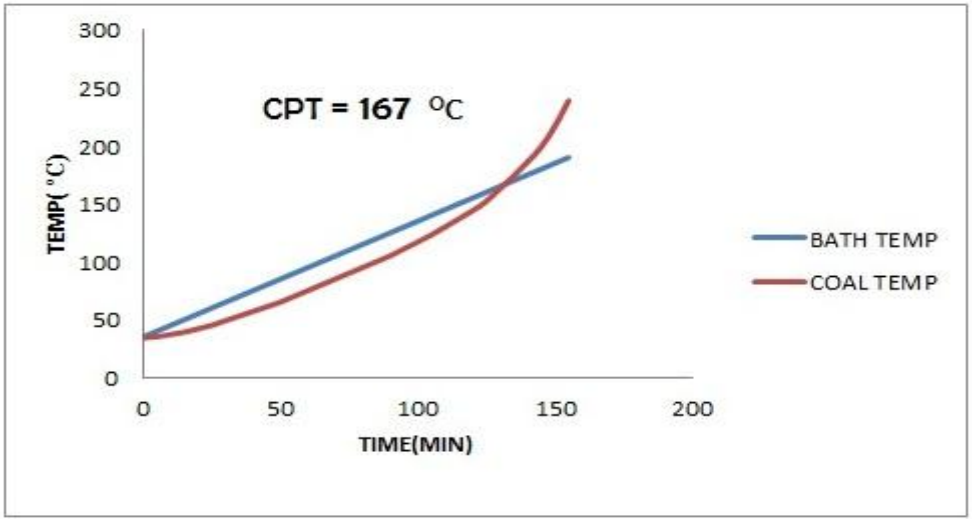

Fig. B-5 CPT curve of CCL-5

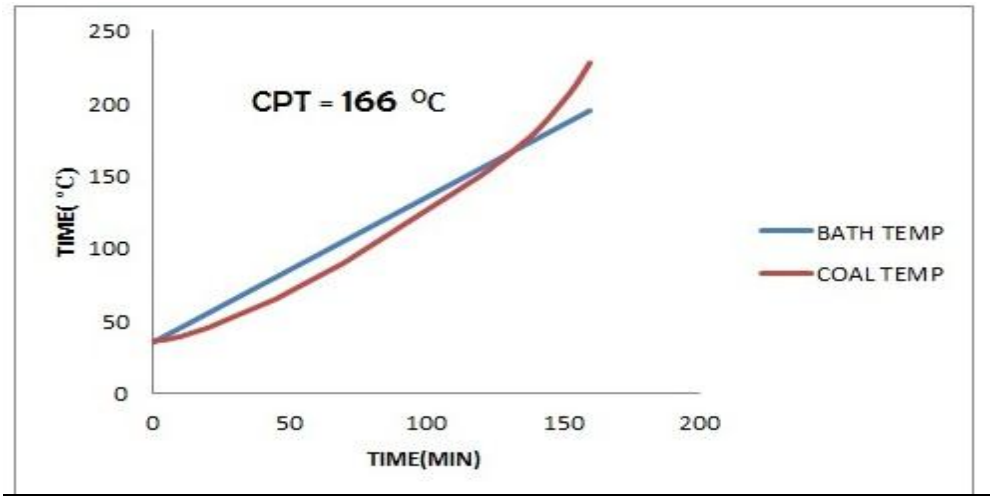

Fig. B-6 CPT curve of MCL-1

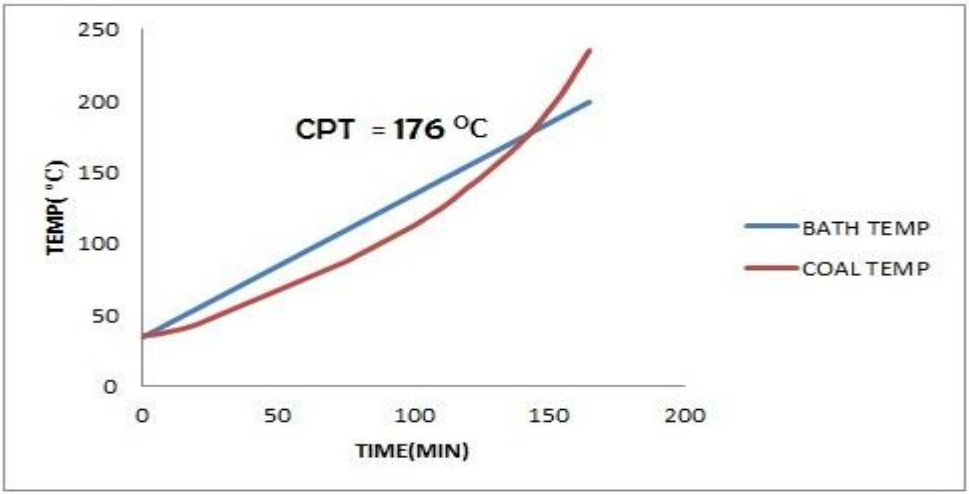

Fig. B-7 CPT curve of MCL-2

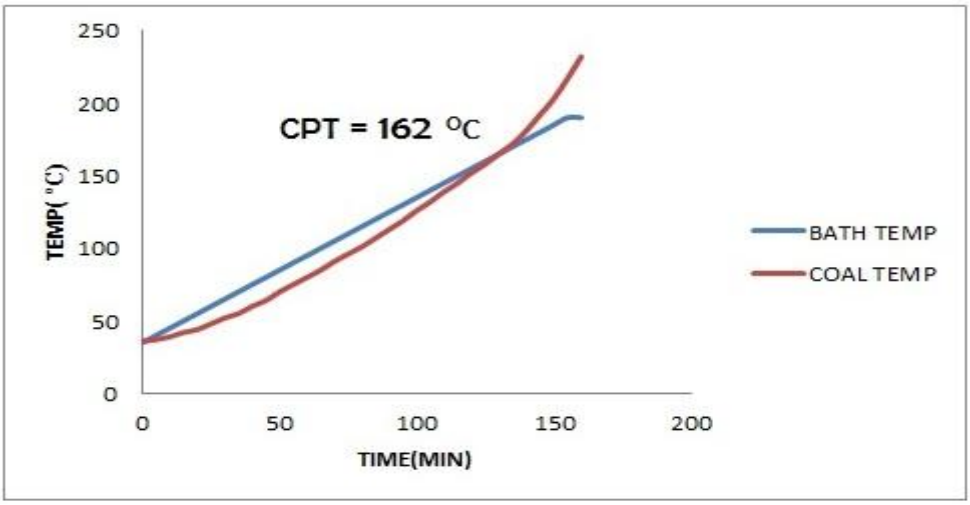

Fig. B-8 CPT curve of MCL-3

APPENDIX-3

OLPINSKI INDEX CURVES

The graphical presentations of Olpinski index of all the coal samples are shown in the following figures:

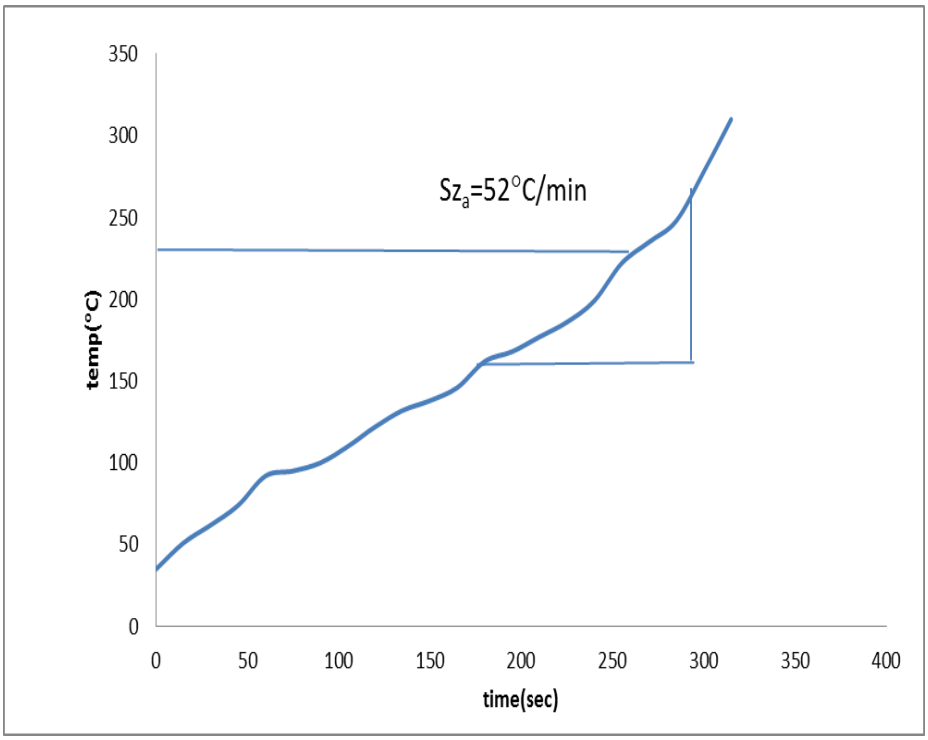

Fig. C-1 Olpinski curve of CCL-1

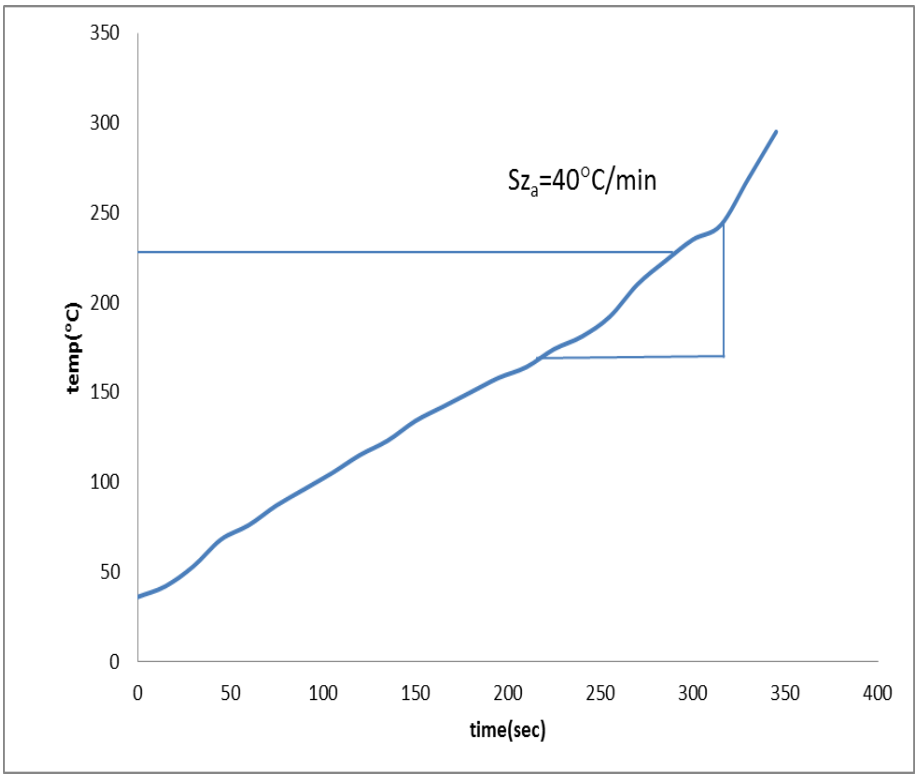

Fig. C-2 Olpinski curve of CCL-3

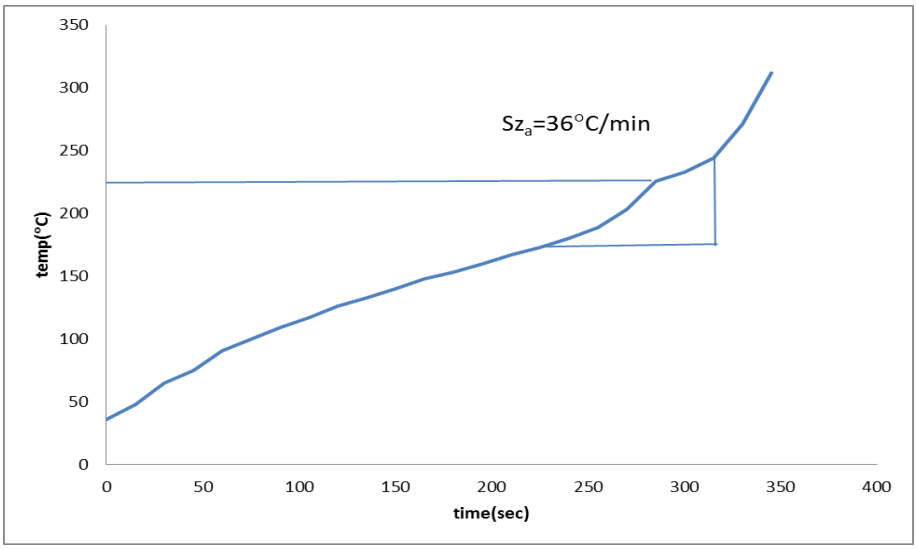

Fig. C-3 Olpinski curve of CCL-3

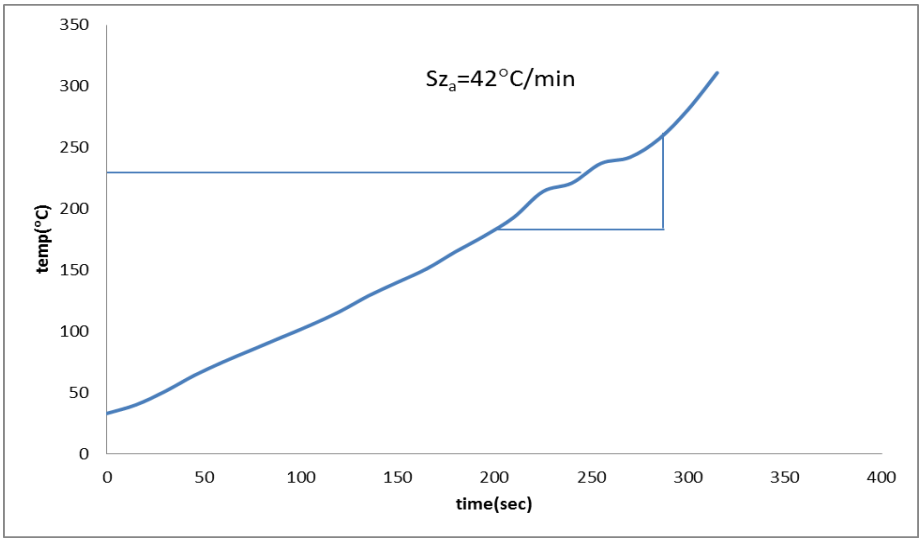

Fig.C-4 Olpinski curve of CCL-4

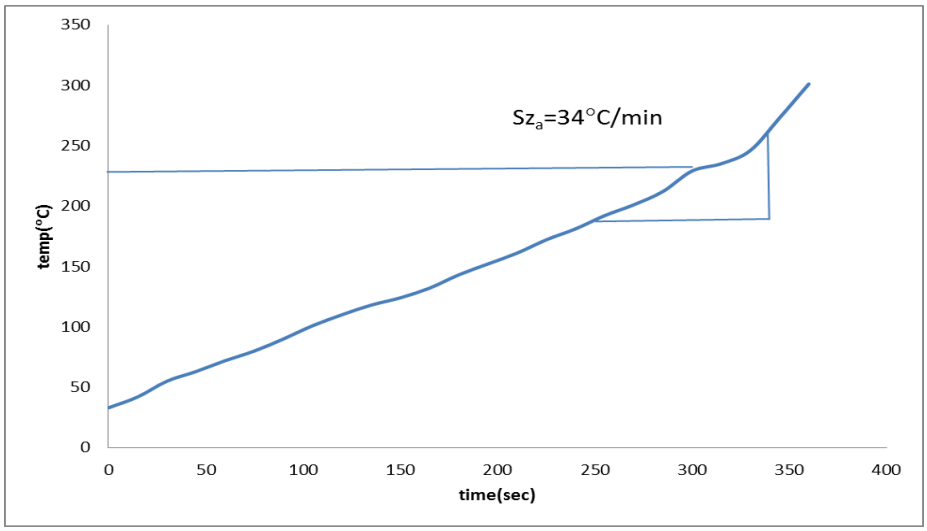

Fig. C-5 Olpinski curve of CCL-5

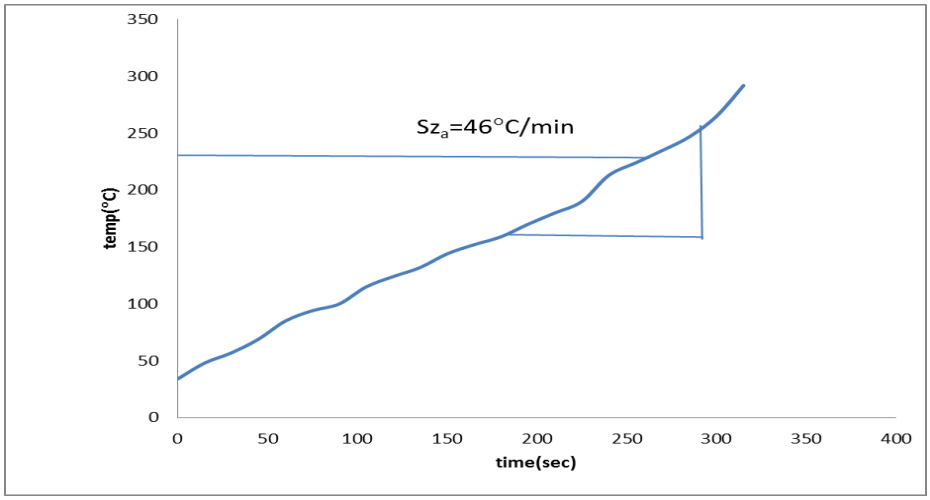

Fig. C-6 Olpinski curve of MCL-1

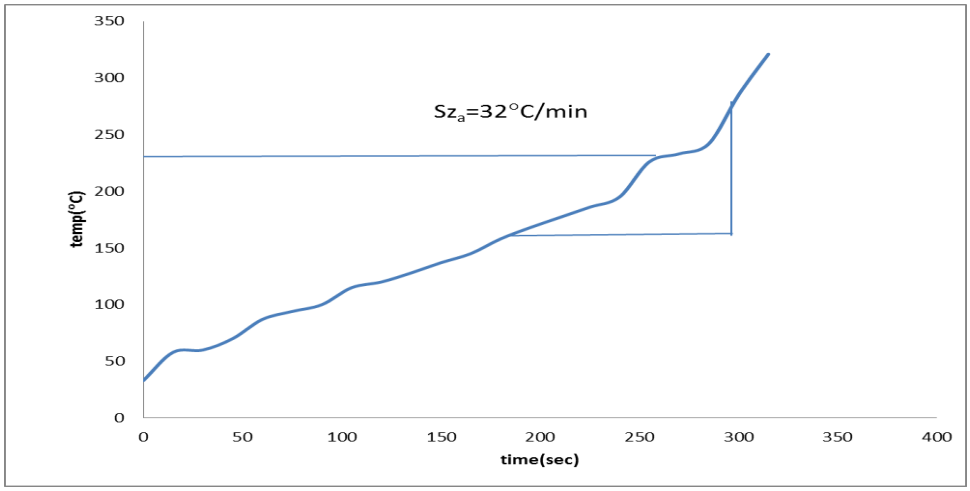

Fig. C-7 Olpinski curve of MCL-2

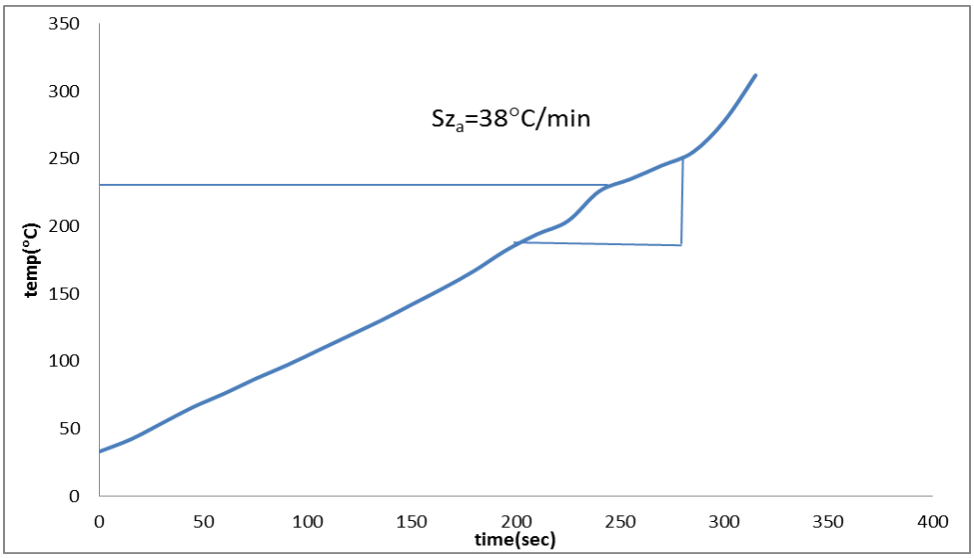

Fig. C-8 Olpinski curve of MCL-3

APPENDIX-4

DTA CURVES

The differential thermal analysis curves obtained from all the collected coal samples are shown in the following figures:

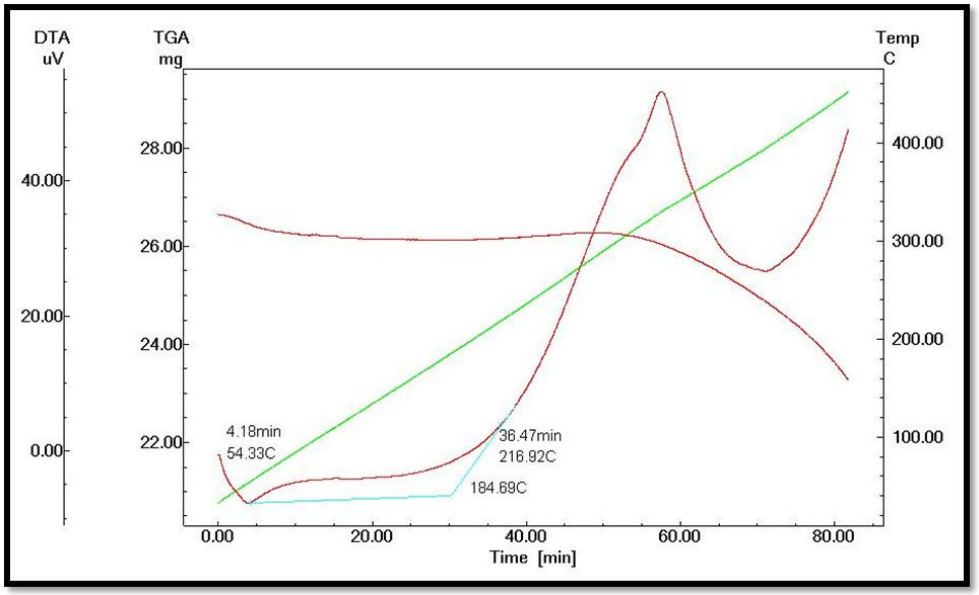

Fig. D-1 DTA curve of CCL-1

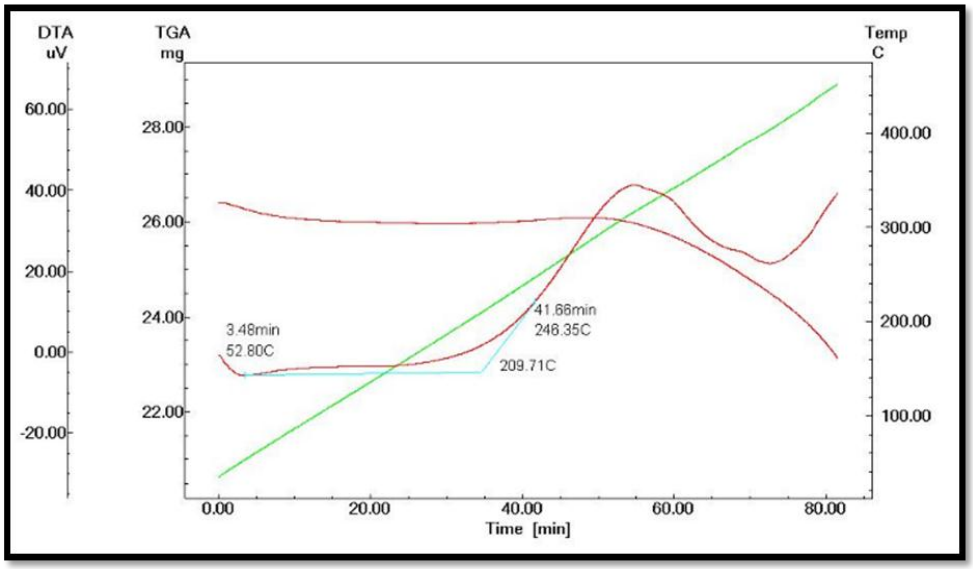

Fig. D-2 DTA curve of CCL-2

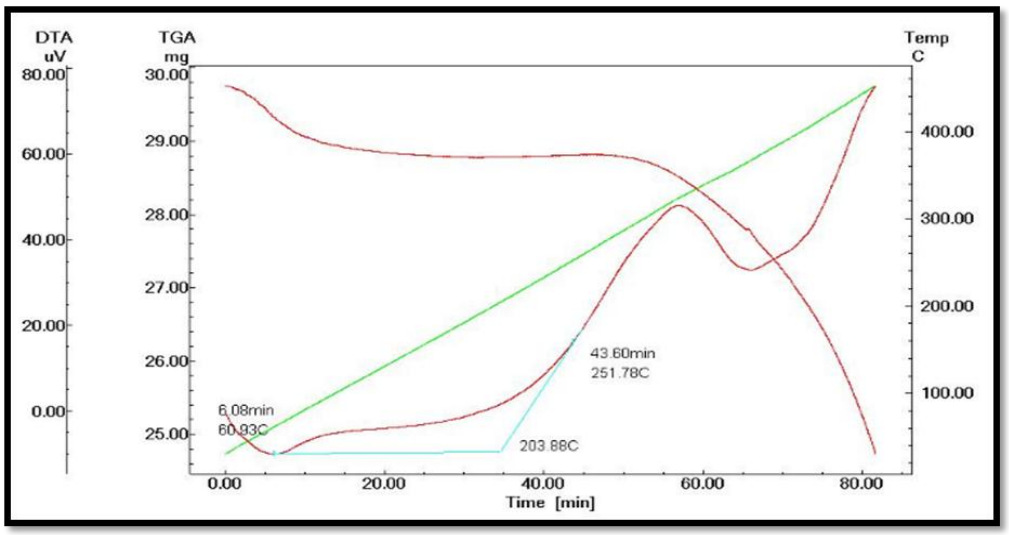

Fig. D-3 DTA curve of CCL-3

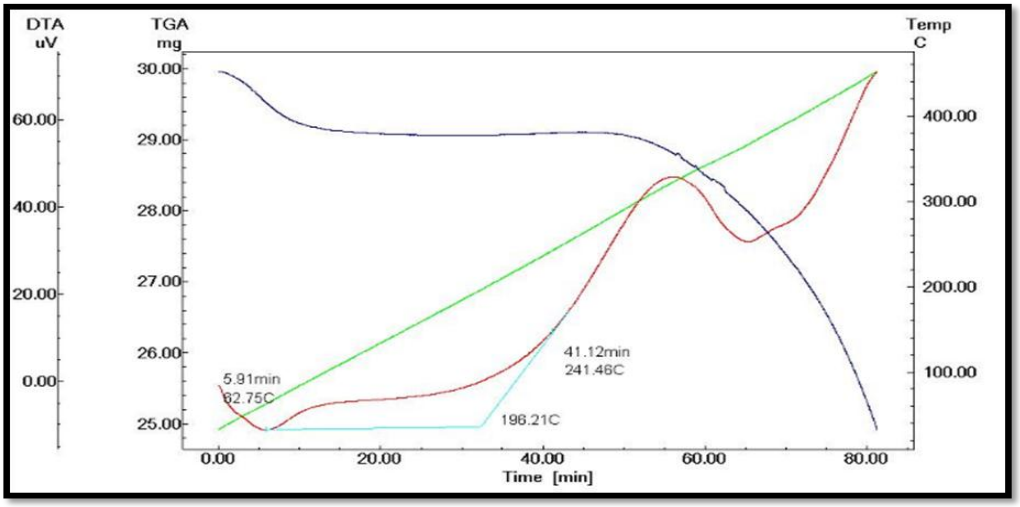

Fig. D-4 Olpinski curve of CCL-4

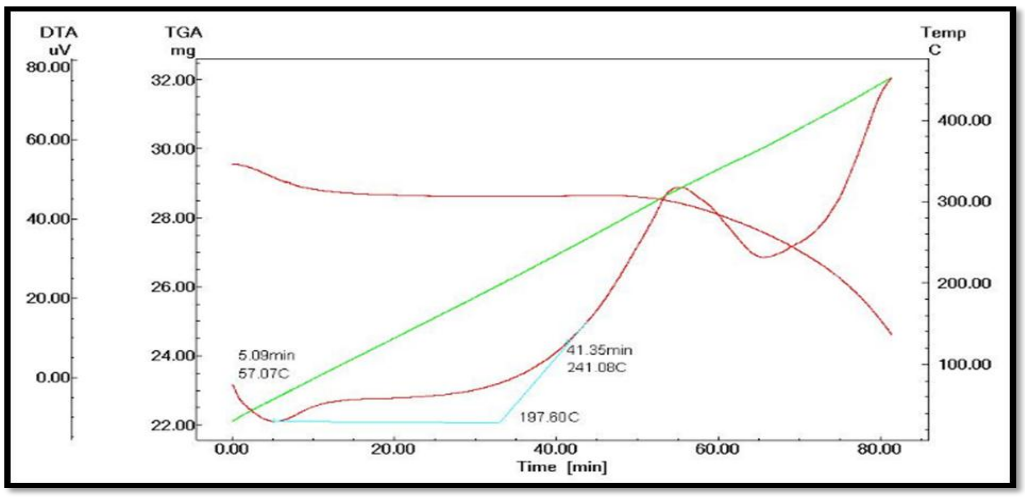

Fig. D-5 DTA curve of CCL-5

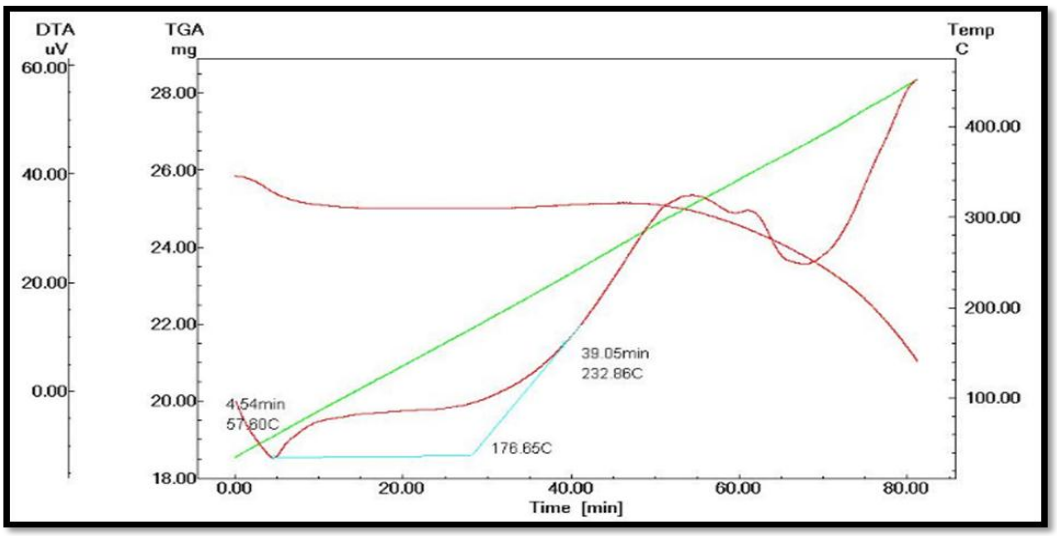

Fig. D-6 DTA curve of MCL-1

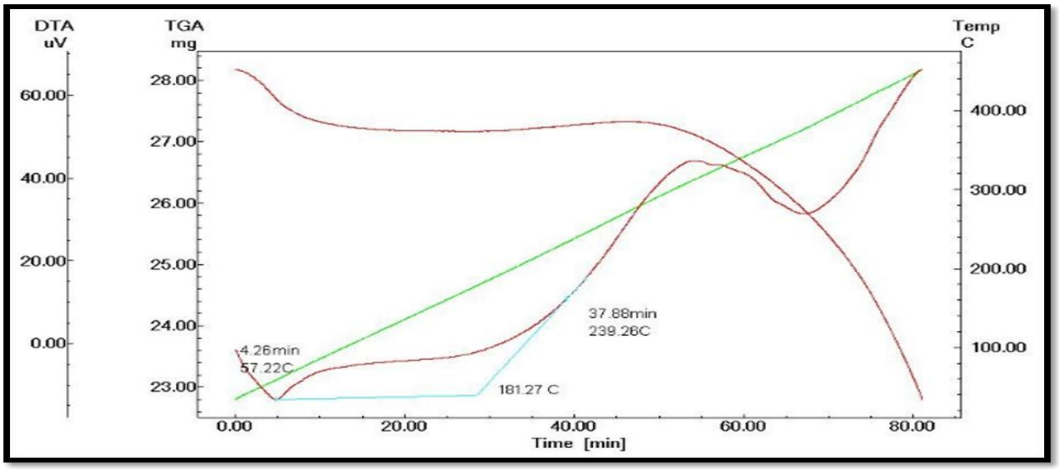

Fig. D-7 DTA curve of MCL-2

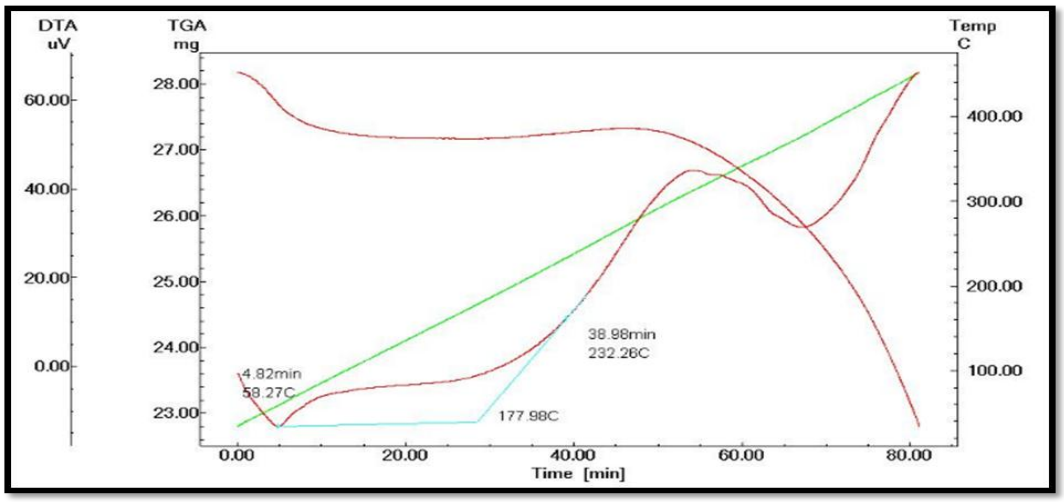

Fig. D-8 DTA curve of MCL-3

APENDIX-5

CORRELATION CURVES

The correlation analysis between the parameters of proximate analysis and susceptibility indices are shown in the following figures:

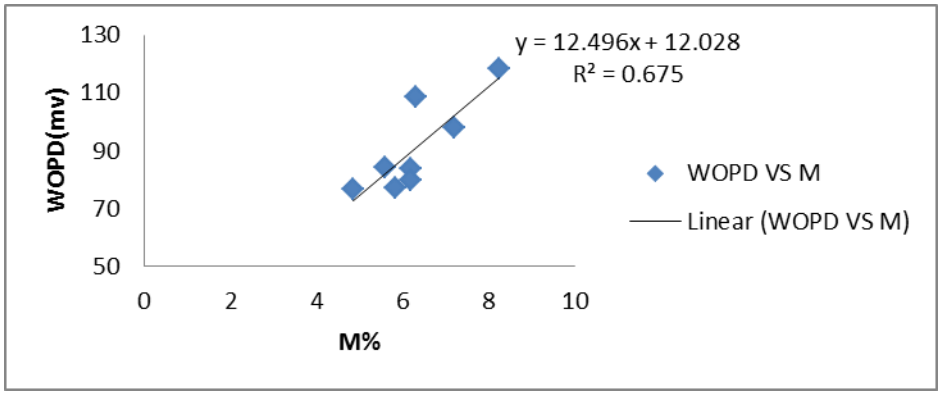

Fig. E-1 Correlation curve between Moisture and WOPD

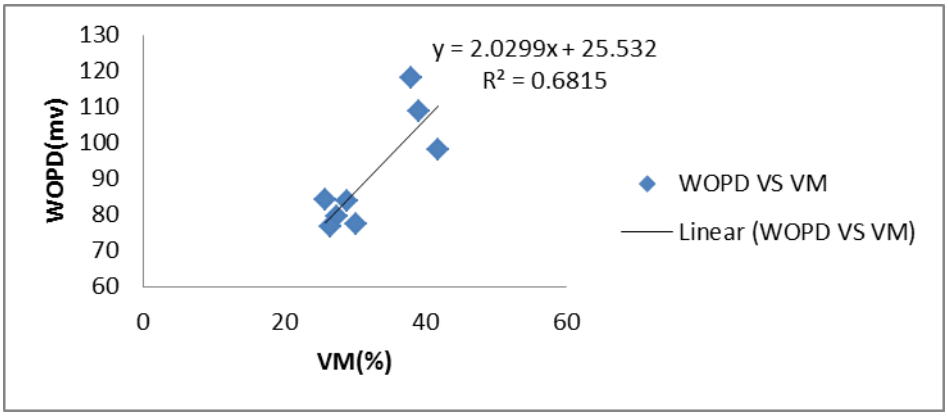

Fig. E-2 Correlation curve between volatile matter and WOPD

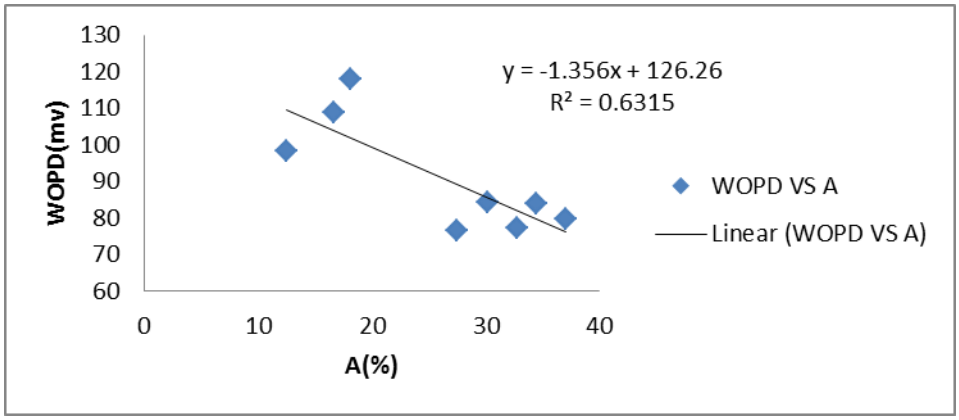

Fig. E-3 Correlation curve between ash content and WOPD

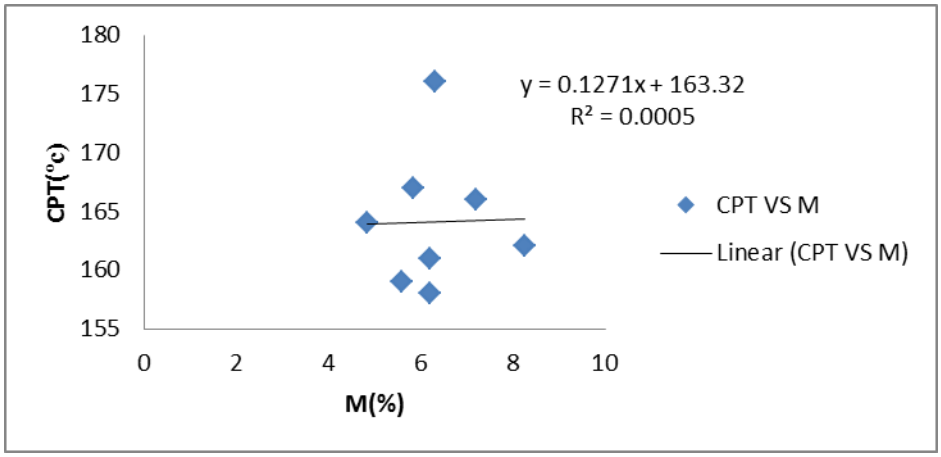

Fig. E-4 Correlation curve between moisture and CPT

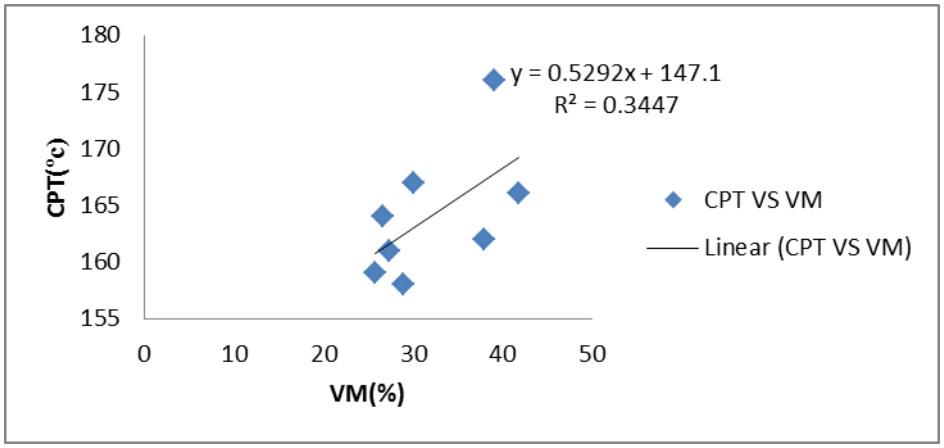

Fig. E-5 Correlation curve between volatile matter and CPT

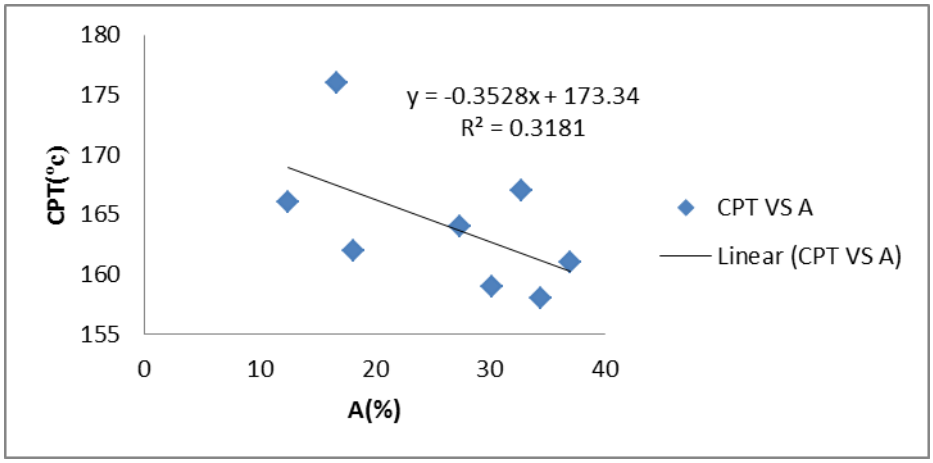

Fig. E-6 Correlation curve between ash content and CPT

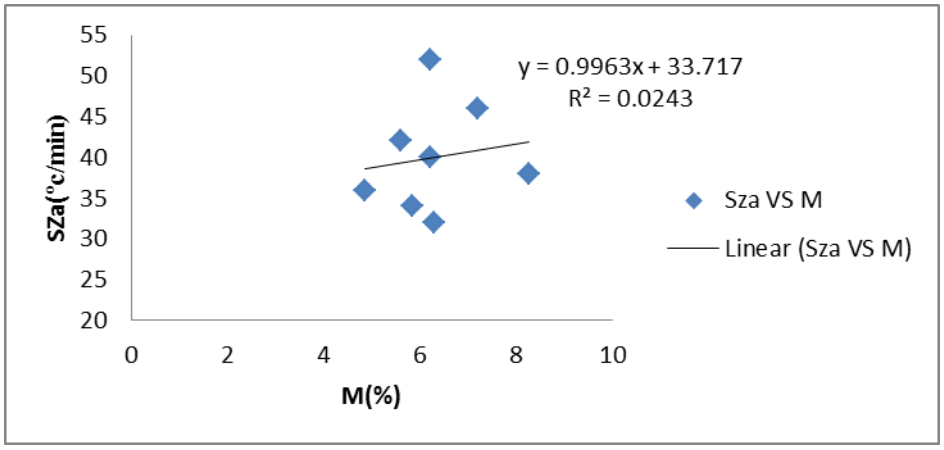

Fig. E-7 Correlation curve between moisture and Sz_a

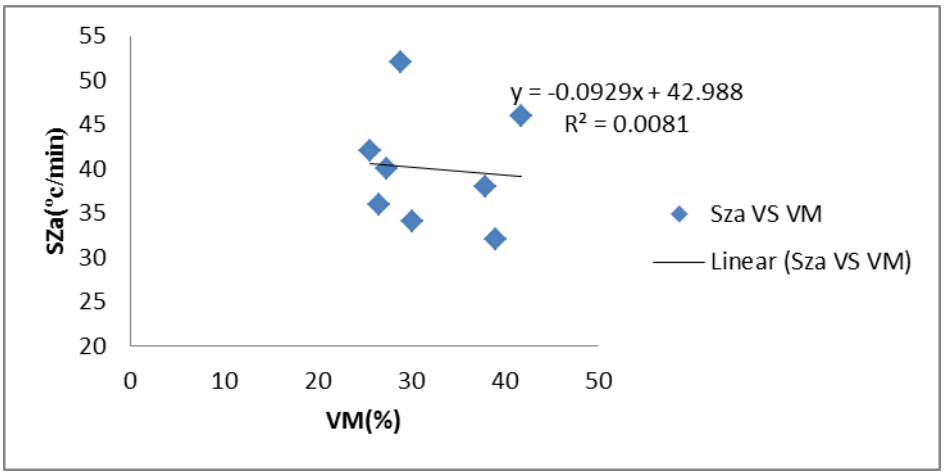

Fig. E-8 Correlation curve between volatile matter and Sz_a

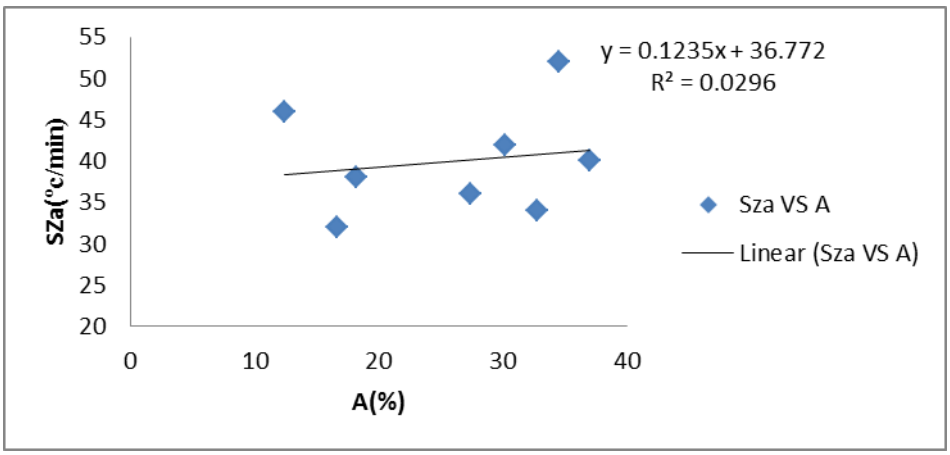

Fig. E-9 Correlation curve between ash and Sz_a

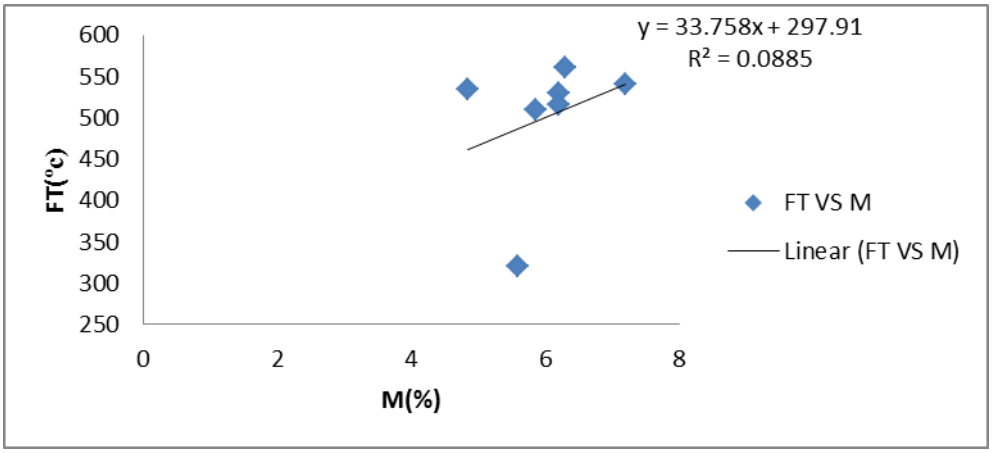

Fig. E-10 Correlation curve between moisture and flammability temperature

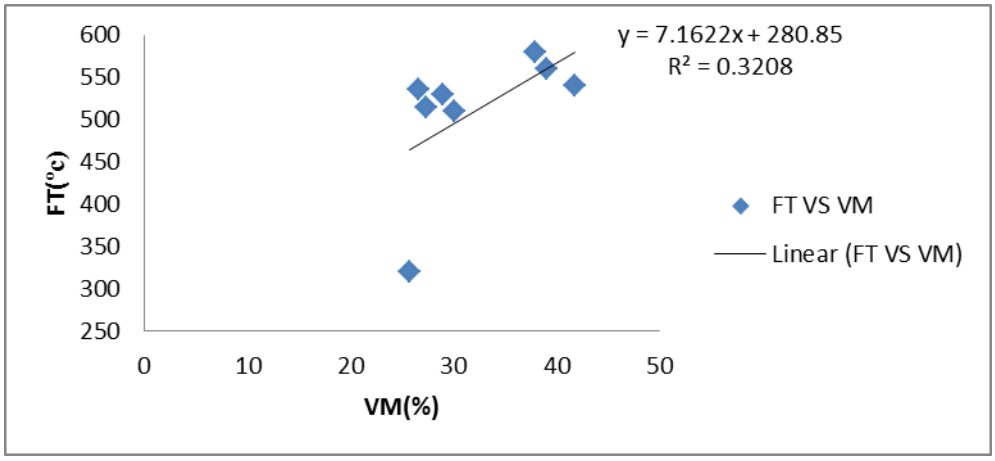

Fig. E-11 Correlation curve between volatile matter and flammability temperature

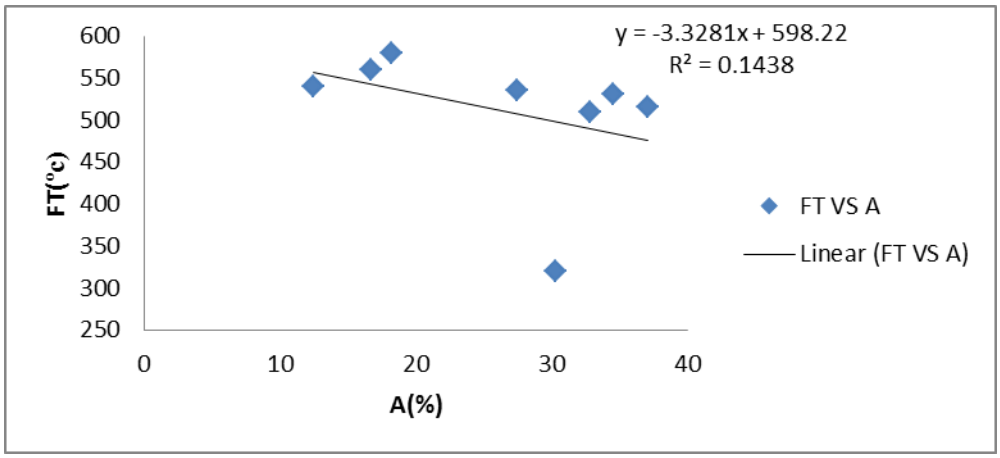

Fig. E-12 Correlation curve between ash and flammability temperature

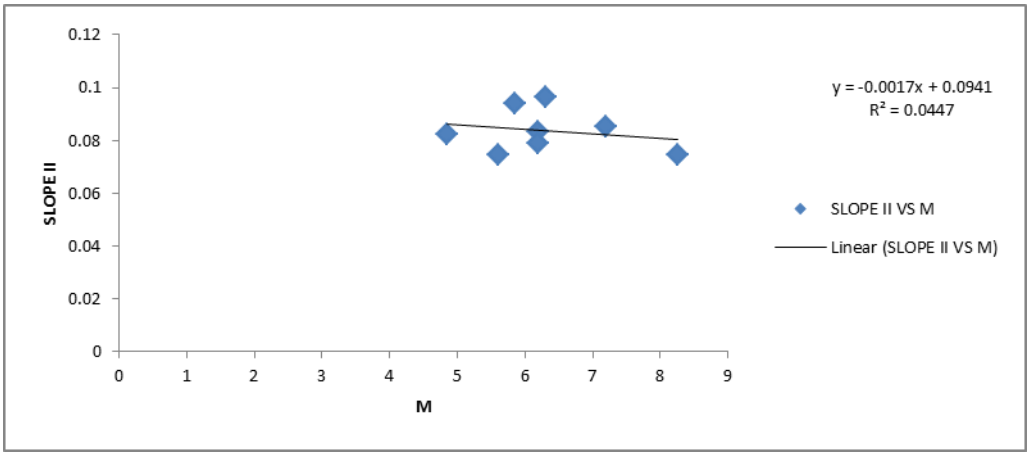

Fig. E-13 Correlation curve between moisture and slope II

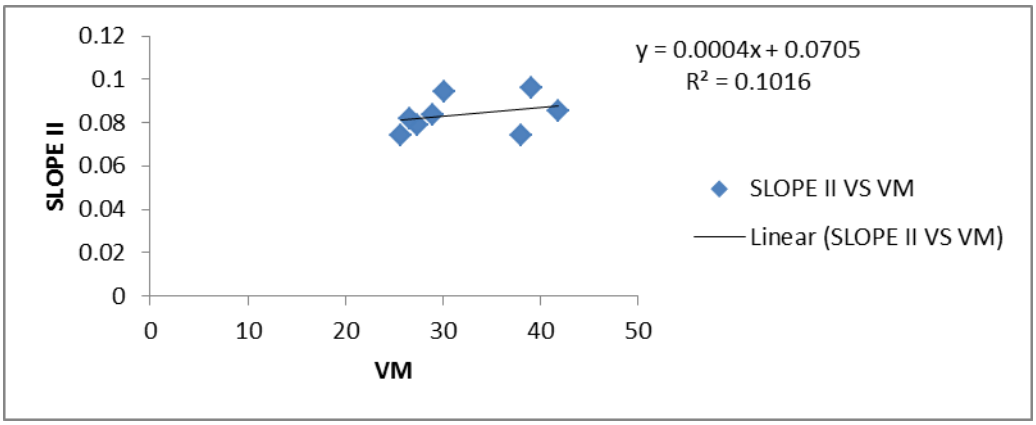

Fig. E-14 Correlation curve between volatile matter and slope II

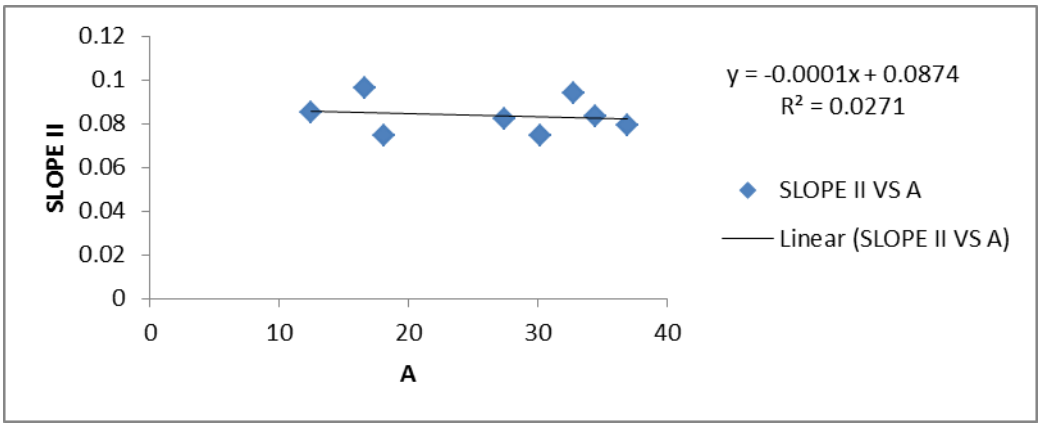

Fig. E-15 Correlation curve between ash content and slope II

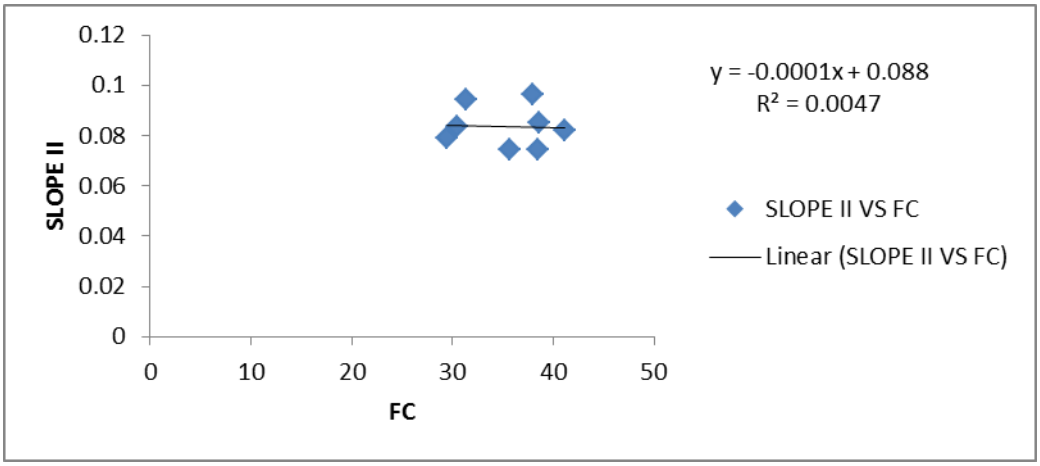

Fig. E-16 Correlation curve between fixed carbon and slope II

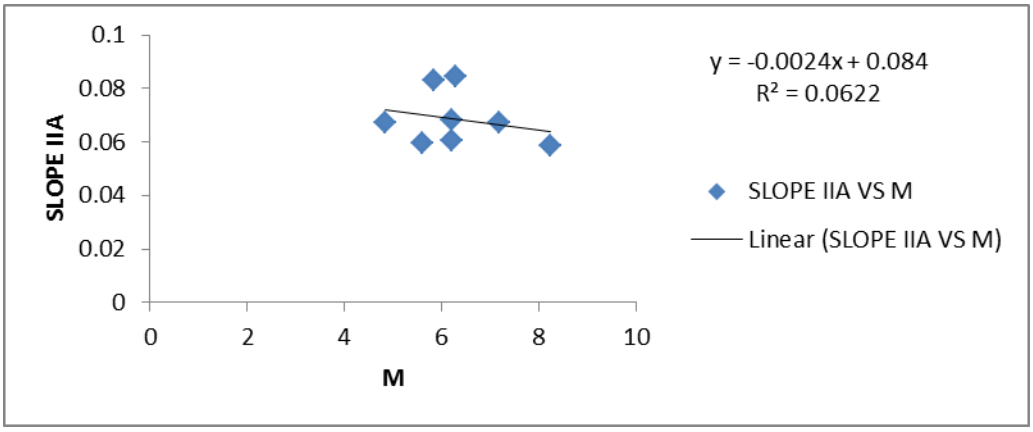

Fig. E-17 Correlation curve between moisture and slope IIA

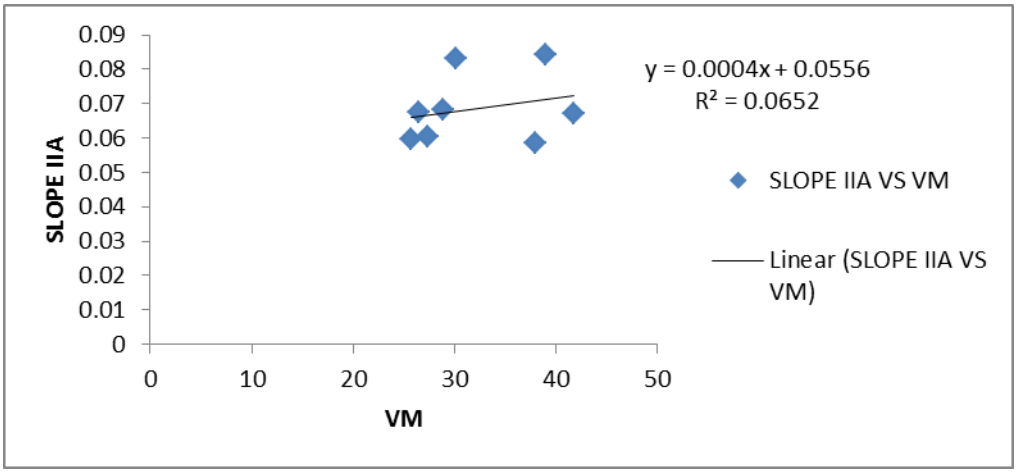

Fig. E-18 Correlation curve between volatile matter and slope IIA

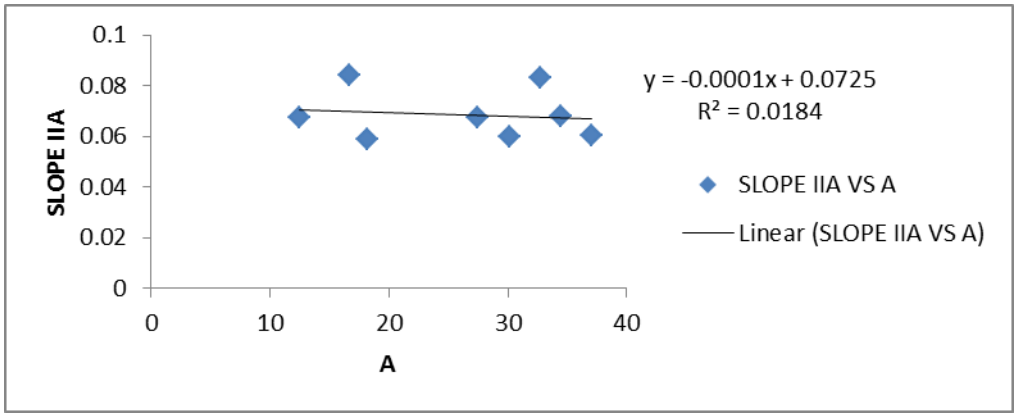

Fig. E-19 Correlation curve between ash and slope IIA

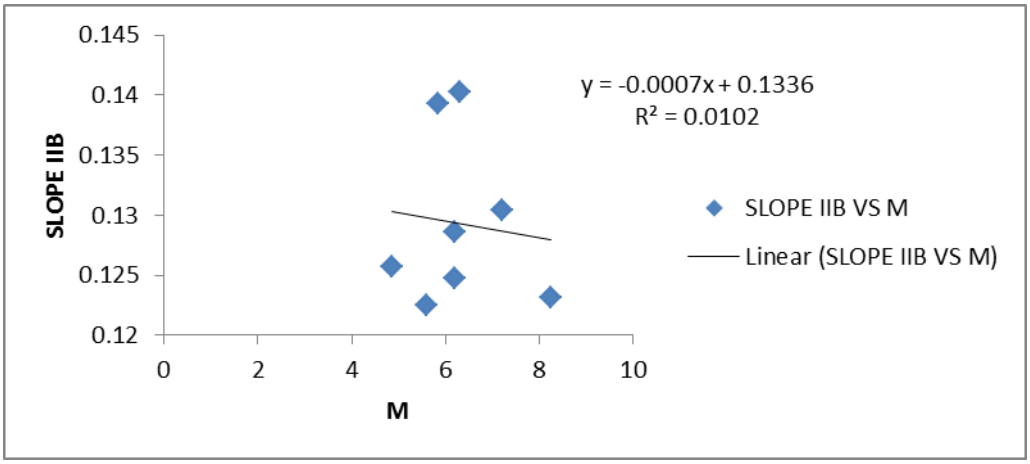

Fig. E-20 Correlation curve between moisture and slope IIB

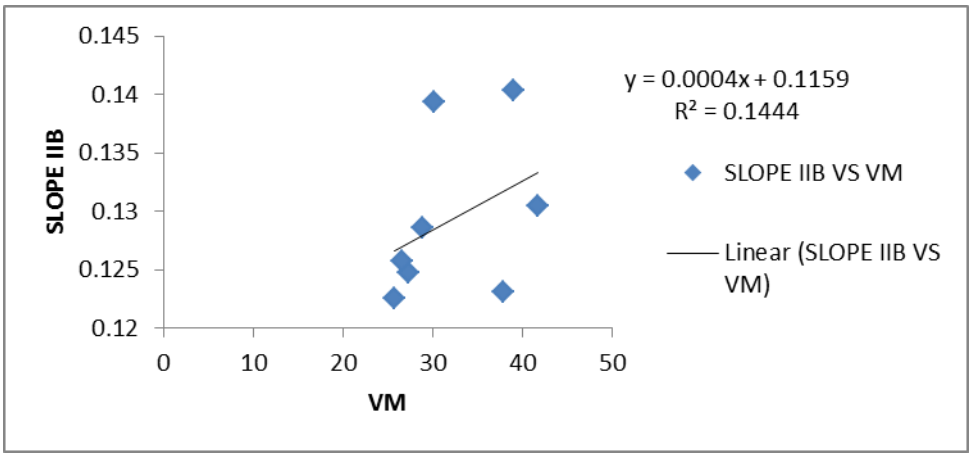

Fig. E-21 Correlation curve between volatile matter and slope IIB